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Issue of Journal. The Journal is published in six bi-monthly parts per volume, viz., February, April, June, August, October and December. A brochure giving the History, Regulations and List of Members of the Institution was published in January, 1923, and the revised List of Members will be published periodically.

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English equivalents of foreign weights and measures should be given, and it is suggested that a bibliography of the subject dealt with be appended.

All papers and articles submitted are assumed to be original communications unless otherwise stated, in which case reference to the previous publication should be given.

Members are invited to submit papers to be read at the General Meetings of the Institution, and are specially asked to forward articles for publication in the Journal.

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Bibliography and Abstracts. A bibliography of current literature is published with each issue of the Journal, together with abstracts of the more important articles and patent specifications, this supplement being paged separately to the Transactions. In order to make this section of the maximum value, members are requested to send any matter which may have been inadvertently overlooked to the editor.

Medal for Student Members. A medal and a prize of five guineas will be awarded annually by the Council to that Student Member of the Institution who shall, in their opinion, have presented a paper of outstanding merit during the year.

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Library. The Institution's Library may be consulted between the hours of 11 a.m. and 4 p.m. daily. The additions made to the current and standard literature on petroleum and allied substances, since the publication of the last Journal, are :—

From the Publishers (for review) :—
The Genesis of Petroleum. By Percy E. Spielmann.

From the International Institute Branch, Dept. Agriculture, Ottawa :—
List of Publications added to Library, No. 1, 1923.

From A. Rauch :—
La Roumanie Economique.

From the Memoirs of the Geological Survey :—
Summary of Progress of the Geological Survey of Great Britain and the Museum of Practical Geology for 1922.

From the Dominion Bureau of Statistics, Canada :—
Revised Statistics on the Mineral Production of Canada, 1922, No. 14, Crude Petroleum.

From the British Engineering Standards Association :—

Complete set of Specifications to date.

From the Imperial Mineral Resources Bureau :—

The Mineral Industry of the British Empire and Foreign Countries. Statistics, 1919-1921, for Copper, Petroleum and Allied Products, Silver, Gold, Coal, Coke and By-Products, Lead, Tin, Zinc, Aluminium (including Bauxite and Cryolite), Arsenic and Diatomaceous Earth.

From the Diesel Engine Users' Association :—

Report on a Broken Crankshaft on a Diesel Engine at Bridgetown, Barbados. By W. C. Shettle, A.M.I.C.E., and Report of Discussion.

Comparative Running Costs of Steam and Diesel driven Pumping Plants. By K. B. Woodd Smith and T. D. Key, A.M.I.C.E., and Report of Discussion.

From G. W. E. Gibson :—

Considerations pratiques sur le pompage des Petroles.

From the Iron and Steel Institute :—

Journal, Vol. CVII., No. 1, 1923.

From B. J. Ellis :—

Notes on American Oil Producing Methods made a Tour of the Fields, February to August, 1923.

From the U.S. Bureau of Mines, Dept. of the Interior :—

Serial No. 2530. Lifting costs at Oil Well Properties. By H. C. George.

Bulletin 744. The Lime Belt of Massachusetts and parts of Eastern New York and Western Connecticut.

Bulletin 751-B. Progress report of a subsurface study of the Pershing Oil and Gas Field, Osage County, Oklahoma.

Natural Gas in 1919-1921. By R. S. McBride and E. G. Sievers.

Bulletin 748. The Twentymile Park District of the Yampa Coal Field, Routt County, Colorado.

Bulletin 709. Triangulation and Primary Traverse, 1916-1918.

Bulletin 689. Boundaries, Areas, Geographic Centers and Altitudes of the United States and the several States.

Professional Paper 132-A. Rock formations in the Colorado Plateau of South-Eastern Utah and Northern Arizona.

From the Dept. of Mines, Geological Survey of Canada :—

Summary Reports 1922, Part A, No. 1989; Part D, 1922, No. 1990.

Memoir 133. The Southern Part of the Sydney Coal Field, Nova Scotia.

Memoir 134. Brockville-Mallorytown Map-area, Ontario.

The Council would be grateful to members who are authors of books on petroleum or on allied substances if they would kindly present a copy of their works to the Library of the Institution, and for review in the Journal.

PERSONAL NOTES OF MEMBERS AND SPECIAL NOTICES.

It is suggested that members send information regarding their movements to the Secretary, for insertion under this heading.

Mr. H. E. Morris has returned from Burma.

Mr. Albert Millar is now back in Galicia.

Mr. R. Van Sickle is now in England.

Mr. E. J. Thompson has left England for Durban, Natal, South Africa.

Mr. A. P. Catherall has returned to Trinidad.

Mr. J. A. Child has returned from North-West Peru.

Dr. J. A. L. Henderson has left Canada for England.

Mr. F. E. Keep is leaving India and should reach England during January.

Mr. Foxall-Smedley is on his way to Burma.

Mr. C. O. Graham is now in Kobe, Japan.

Major R. W. Barnett was returned as Member of Parliament for the South-West St. Pancras constituency at the recent General Election.

Dr. Arthur Wade is leaving early in January to carry out a reconnaisance of the Northern Territory of Australia on behalf of the Commonwealth Government of Australia.

We are indebted to the Editor of *Engineering* for his courtesy in loaning us the blocks of the illustrations of Mr. Millar's paper in this Journal.

We desire to express our thanks to Mr. F. B. Plummer, M.Am.Inst.Min.Eng., M.Am.Assoc.Pet.Geol. (Member), of Houston, Texas, for his account of the International Petroleum Exposition and Congress, held at Tulsa, Oklahoma, October 8th to 14th, 1923, published in this Journal.

CHARLES FREDERIC MABERY.

Prior to his first association with the petroleum industry in 1887, Professor Mabery was closely associated with the Cowles brothers in the development of their internally-heated electric furnace.

As a graduate of chemistry in the Lawrence Scientific School, he was one of the few to receive the degree of Doctor of Science in the Havard graduate school. His first position was as assistant chemist at Havard College, and for ten years as Director of the Havard Summer School in Chemistry for teachers. In 1883 he became professor of chemistry at the Case School of Applied Science, a position which he occupied until 1911, when ill-health caused his retirement. Since then he has spent as much time as his health would permit on research work.

He has chiefly occupied his time with research into the constitution and action of lubricants, and we are glad to be able to publish, through the courtesy of the *Journal of Industrial and Engineering Chemistry*, a paper by Professor Mabery on the "Lubricant and Asphaltic Hydrocarbons in Petroleum."

PETROLEUM RESEARCH AND REFINERY.

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THE INSTITUTION OF PETROLEUM TECHNOLOGISTS.

THE SEVENTY-THIRD GENERAL MEETING of the Institution of Petroleum Technologists was held at the House of the Royal Society of Arts on Tuesday, November 13th, 1923, Mr. H. BARRINGER, M.Inst.C.E., M.I.Mech.E., M.I.N.A. (President) occupying the Chair.

The following paper was read :—

Galician-Canadian Pole Tool Fishing Methods.

By ALBERT MILLAR, A.M.I.Mech. E. (Member).

"FISHING" in oil well nomenclature covers a great variety of work, and does not merely represent the recovery of lost tools as is often thought to be the case. Sidetracking of tools or casing also comes under the heading of "Fishing."

Of all practical oilfield work fishing engages the greatest amount of skill and attention combined with an unlimited amount of patience and ingenuity. In all fishing operations "touch" plays a most important part, and this sense can only be acquired by long practice, as does also the knack of constructing suitable fishing tools to suit individual cases. Some smiths possess a really marvellous faculty for forging a tool of the exact shape and dimensions required, so that one would almost think he had demonstrated on the article to be fished for. In most fishing cases the smith plays as equally important a *rôle* as the driller, therefore it is most essential that harmony should prevail between the drilling staff and the smithy department.

A good fisherman must be imaginative and be able to reconstruct the probable position in the borehole in his mind, and to foresee that if a certain instrument is let in, just how that instrument is going to function. When a complicated fishing job is in progress it will be found to be a very good plan, before commencing operations, to draw to scale the probable position in the borehole, taking, as a basis, diameter of casing, dimensions of lost tools, measurements of depths, and impressions obtained. This enables one to form a much better idea as to what has happened, the type of tool to be selected, and its construction. As work proceeds and the position becomes clearer, revised sketches should be made and the dimensions

of the fishing tool being used should be noted by the side of the sketch. In the event of the misfortune occurring to lose a tool whilst fishing, the situation can be immediately dealt with.

It should always be borne in mind, that at the best, fishing is carried on in the dark, therefore it is most important that every possible detail should be noted and recorded on paper instead of trusting to memory. If, in addition to making sketches, the possible position in the borehole is reconstructed on the surface, a still better idea as to the best method of procedure will be obtained. This is done by taking a length of casing of the diameter being fished in, and duplicating the position in the borehole as far as is known from impressions taken, this also facilitating the construction of the type of fishing tool required.

The majority of fishing cases are initially caused through carelessness or lack of proper foresight, and are avoidable to a very considerable extent. For instance: running drilling poles and ropes after they have reached a state of fatigue; using tools of which the joints have become worn; or badly shaped bits causing the pins to break; or screwing up casing too tightly, thereby putting unnecessary strain on the boxes, etc. These are frequent errors, and are apt to lead to more or less complicated fishing. A great deal depends on the driller and his personnel exercising proper care and forethought, but however careful they may be, it is advisable that the engineer-in-charge should make a periodical examination of all tools, joints, ropes, casing, etc.

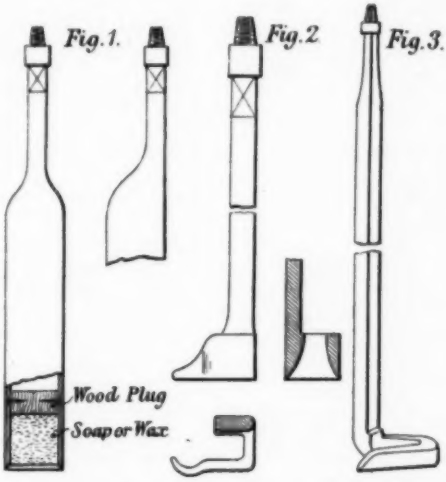
If all those engaged in the actual drilling of wells would not lose sight of the important fact that iron and steel can be mishandled just like any other material, many a fishing trouble, with its attendant worry and expense, would be avoided. Perhaps one of the most frequent causes of trouble arises from badly designed and cheap tools, which are necessarily also of inferior quality. The greatest care and consideration should be given to the selection of plant and tools, no matter whether operations are contemplated on a large or small scale. The best plan is to leave the ordering of equipment to those who have had good practical oilfield experience and are consequently the most competent to select the right kind of tools and material for the job, but how often is this the case, especially in pioneer prospecting work.

It is impossible to eliminate accidents entirely, but it is possible to lessen the chance of their occurring to a very great extent; yet in spite of every care being exercised, accidents happen. In the majority of cases it is not the original accident which gives subsequent trouble, but complications arising from overhaste. It is therefore important that every move should be most carefully considered from every point of view: one false step may be the

cause of a great deal of trouble and anxiety, and eventually the abandonment of a well after months of tedious and costly fishing.

The engineer-in-charge, as well as the driller, should know the exact length, size, and weight of the string of tools in use, as well as the length and other measurements of each piece of casing in sequence comprising the working column. Should the drill pin break, the length from cutting edge to shoulder is ascertained at once, and in the event of casing pulling apart or breaking off, the length of the piece, or number of pieces contained in that part of the column which has parted, is precisely known.

Before commencing actual fishing operations the distance between the top of the casing and the object being fished for should be accurately measured and checked; time taken in measuring is not time lost but time gained in the long run. An impression block (Fig. 1) can be run at the same time as measurements are being



taken. For this purpose the impression socket (containing from 6 in. to 10 in. of soap or wax) should be of as large a diameter as the casing will allow, and from 5 ft. to 6 ft. long. This socket can also be made to work eccentrically, and should be run until a well-defined imprint has been obtained, as on this depends the type of tool to be employed. Only in the most simple of cases is it permissible to lower in a socket or other fishing tool before the impression block has been run. It is, however, by far the better and safer course not to commence fishing before taking an impression and accurately

measuring the distance. The right kind of tool has then to be selected to cope with the situation. Should this not be in stock, or cannot be constructed in the well smithy, then it is advisable to suspend operations until it has been provided. Makeshift tools are dangerous, and should not be used.

Lost Tools.—Little or no difficulty is experienced in the recovery of tools which have become unscrewed during drilling, *except* in the following cases :—

When the driller has not detected that his bit has become detached and continues drilling on it, then either of two things are liable to happen. If he is drilling in soft strata, the lost tool will be driven off to the side ; should however the formation be hard, a head will be formed on the lost tool, which may attain such dimensions that there will be no room left to pass a socket over it.

When the bit has been driven off into the wall, a hook instrument (wall hook) should be used to catch hold of, and to pull, the lost tool into the centre of the borehole to enable it to be grasped by the socket. The shape and dimensions of the hook vary with individual cases, according to the size of the borehole and the tool to be pulled into position (Figs. 2 and 3).

In cases where a head has been formed by continued drilling on the bit after it has become detached, the best procedure is to file the head off. This work can be accomplished in a few hours, and any good smith can construct the file or rasp tool. The width of this file or rasp is generally from 4 in. to 7 in. wide and from 6 ft. to 12 ft. long, tapering gradually and being slightly smaller at the bottom. The construction of the file depends on the size of the hole, tool, and work to be done ; the surface of the rasp tool is generally concave. This file or rasp is a most handy tool, and accomplishes a great deal of work in a short space of time. It should be constructed from good material, which is not brittle, and "springiness" is a most essential factor. After the lost bit or sinker has been pulled into the centre, or the head filed off, a socket may be lowered in. The best type for this work is the split taper socket (horn socket), Fig. 4, fitted with one or more dogs. The bottom of the socket should be cut off at an angle and the point or apex should be to the inside. It is possible to recover objects lying to one side of the borehole with this tool.

When, as is sometimes the case, the tools defy recovery, they can be sidetracked—*i.e.*, drilled off to the side. The best tool to use for this purpose is the "hoof bit" dressed at an angle of 45°. Before drilling off to one side is commenced, a filling of hard rock or broken-up cast iron, such as firebars, etc., should be tamped in, this retarding progress, and giving the bit a better chance of thrust-

ing the tool "off." It sometimes happens that bent tools are straightened out by this process and can be withdrawn through the casing in the usual manner.

Stuck or Lodged Tools.—Tools occasionally become stuck during drilling and are left behind. The principal cause of this is: sticky clays, heaving clays, sands, heavy falls of cavings, a boulder falling

FIG. 4.

in, or a slab of rock sliding across the borehole. In cases of this kind, the moment it is noticed that the tools are becoming or have become stuck, upward and downward jarring should at once be tried. If this is not effective in freeing the tools, the drilling rope should be severed as near the tools as possible, or the drilling poles unscrewed. The next thing is to loosen up the tool which has become seized; this is accomplished either by means of a spud, or a flat chisel bit welded directly on to a flexible sinker bar. When the spud has been worked well around and down to the bottom of

the tools, a strong tongue socket with jars and a heavy sinker should be lowered in, the tools grasped, and generally after a few upward blows they will be released. A socket which cannot be made to "let go" should never be used. A safer tool, and one which is easily manipulated, is the taper die socket. After this has been securely screwed on, upward and downward jarring can be done and heavy pulls taken. The taper die socket is preferable to any other and should be used whenever possible. Of course this tool can only be used in conjunction with drilling poles, fishing poles or casing. Hollow flush poles are very useful when drilling alongside trapped tools, as the clays, sands, or cavings can be worked out and flushed away at the same time. Tools are more liable to become stuck in some clays than in others. These are generally classed "sticky clays." When this happens it is advisable to remove all water from the borehole with a suction sand-pump and to replace by oil. The clay then loses its adhesive qualities and drilling can be continued without difficulty.

Another method of recovering lost tools or broken-off bits lying to the side, and which cannot be dislodged by the ordinary means, is to drill on with a bit of as large a dimension as possible, until from two to ten feet below the lost tools has been reached, depending on the length of tool to be recovered. After this has been done a wall hool can be lowered in to draw the tool into position, and to guide it into the newly drilled hole, where it will remain in an upright position, so that it can be gripped by a tongue socket in the usual way.

Slightly Bent Tools.—Care has to be taken in the recovery of tools which have become bent; when they are only slightly deformed they can often be jarred into the casing, becoming straightened out during this process, but excessive jarring is liable to damage the casing-shoe and may give subsequent trouble.

Runaway Tools.—More difficulty is generally experienced in the recovery of runaway tools, as they become more or less bent, and trouble is often found in getting them to pass as a unit through the casing. The best plan is to unscrew them one by one—that is, the jars from the sinker and the sinker from the bit—but they are sometimes so deformed that even this is found to be an impossibility. The safest tool to use when unscrewing poles or tools is the taper die socket. Should the tools be of small calibre—say, from 3½ in. to 4½ in.—they may be cut up with a file, or a socket can be lowered in, a good grip taken, and casing with tools can then be pulled together (known as "stripping"). This is a tedious operation but not a risky one, providing proper care is taken.

When pole tools run away the poles generally become bent into all manner of inconceivable shapes, or, breaking off several times,

run down and become wedged together, rendering the fishing job still more complicated. The first thing to do in such cases is to count and measure the number of poles which have been recovered, then to measure the borehole very accurately to ascertain at what depth the top of the lost poles is standing. If measurements show that only a few feet are missing, then it can be safely assumed that the rods are intact as a unit, and the method of procedure is quite simple. After a socket has been let in, and a hold taken, the poles and tools will, in all probability, be withdrawn together. Should the well be a deep one, and it is found that some 600 ft. to 1000 ft. are missing, then one can be pretty sure that the poles are badly twisted, or, what is more likely, that they have broken off after running away, possibly two or three times.

After a good impression has been obtained a tongue socket should be lowered in. When this has gripped upward jarring may be tried for a short time ; should no progress be noted, then on no account should the situation be forced, as further pulling and jarring would only serve to wedge the poles more tightly together. The best thing to do is to unscrew the pole or poles one has got hold of, and after having done this, to lower in an impression block and get a fresh imprint of the situation, then either jar loose if advisable, or unscrew the poles one after another until direct communication can be made on to the tools. Should unscrewing by means of the socket prove impracticable, then a taper die socket should be employed with left-hand fishing poles.

It often happens that the uppermost pole is bent over in the form of a hook, and will not allow the socket being passed over it. In such a case a hook tool (Fig. 8) should be constructed which will pass between the pole and the casing. This is then lowered in, and when in place, a half turn is given to it, and it is then slowly raised until contact has been made ; by jarring and pulling upwards the pole can be straightened out and a socket used. Very often the hook will bring a number of bent poles with it to the surface.

It is possible in practically every instance to fish a well clear of bent and twisted drilling poles, but should an exception prove the rule, then the only thing left to do is to pull the casing and poles simultaneously, providing, of course, the poles and tools are tightly wedged inside the casing and the casing column is free.

Pulling casing and poles together (stripping) is generally a difficult process and is apt to lead to trouble unless care and every precaution is exercised, because, when pulling casing with poles inside, it seldom, if ever, happens that the casing-joints and pole-joints are flush. It will be understood that were the poles perfectly straight this operation would require utmost care, but when the

poles are bent and corkscrewed, or where poles are broken off and are bent and wedged two or three alongside the other, the pulling of the poles and casing simultaneously is a most hazardous operation. The friction hold of the tools wedged in the casing should never be relied upon to retain their grip, preventing their falling through the casing while it is being pulled, especially so if the well is a deep one. The tools and poles should always be held from above by means of a socket or other grappling instrument.

Another means is to pull the casing to a point above where the lost object lies, then to drive the lost poles and tools into the wall of the borehole, using a hoof bit; as drilling proceeds the tools are cased off.

Broken Ropes.—Should the drilling or sand-pump line have parted a rope spear is used, and it depends on the size of the hole

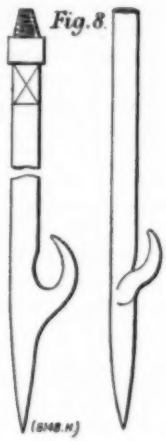


Fig. 8.

whether a single spear, or a double or treble grab, is chosen. The spear is usually from 3 ft. to 5ft. long, and a stout plate should be fixed directly above it of slightly smaller diameter than the casing. This prevents the spear being driven too deeply into the rope, which would most likely form a knot above the spear, thus preventing its withdrawal. When the rope has been grappled, the whole string can be withdrawn at a single operation, providing the tools have not become stuck in the meantime. Should the tools be imprisoned, the rope must be torn out in lengths until a direct communication can be made on to the lost drilling implements or sand-pump.

Sidetracking.—In cases of lost tools or where tools are left in the borehole one alongside the other, the best thing to do is as follows: Take an instance of a set of tools having become stuck; drilling off to the side was commenced, a second string got trapped, and eventually a third, the hole being thus completely plugged. The total length of the three sets of tools can be taken as thirty-five feet or more, and the borehole 6 in. diameter. To overcome the difficulty and enable the well to be deepened past the trapped tools, a good plan is to use a starting bar (Fig. 9) made as follows: About 75 ft. of 5 in. casing is taken of which 20 to 25 ft. should be collapsed in the form of a wedge and then screwed into the remaining lengths, which should be filled in with loose stone and a wooden plug inserted at the bottom. When this is done, lower it into the well until it comes to rest on the obstruction, then withdraw the 6 in. working casing column up to thirty feet or more above the top of the starting bar, and after a filling of stone has been put in, drilling with a hoof bit can be commenced. It will be found that a new hole can be drilled down and past the obstruction, which will not deviate from the vertical by more than six to fourteen inches. Owing to the use of the starting bar, the deflection is so gradual that the 6-inch casing column will follow on quite easily. The time required to accomplish this work depends naturally on the formation being drilled in, but in any case comparatively quick progress can be made. It is always advisable to make starting bars as long as possible.

Casing Accidents.—It sometimes happens that the casing shoe becomes detached, *i.e.*, it either comes unscrewed or it breaks off. There are three ways of overcoming this trouble:—

1. By screwing on again when this is possible.
2. By taking hold of the lost shoe with a casing spear, and pulling out the casing and the detached shoe together.
3. By drilling up the shoe.

Screwing on to the shoe can only be done provided the thread has not been damaged and the shoe is standing upright. After connection has been made, the casing column should be withdrawn in order to control the joint and to ascertain whether the shoe has been properly and securely screwed on.

Should it be decided to take hold with a casing spear, a certain amount of care is necessary in the selection of the type of spear for this job; the "bulldog" type should not be used, but one that can be released at any time as desired (Fig. 10). The manipulation of casing and tools simultaneously calls for much skill and resourcefulness, as previously mentioned.

The most simple and straightforward method is to drill the shoe up, especially if, as in Galician practice, the shoe is only from

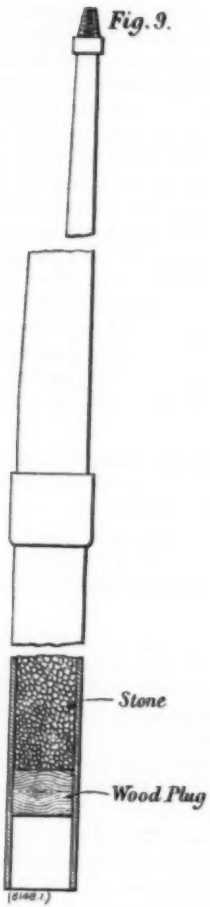


Fig. 9.

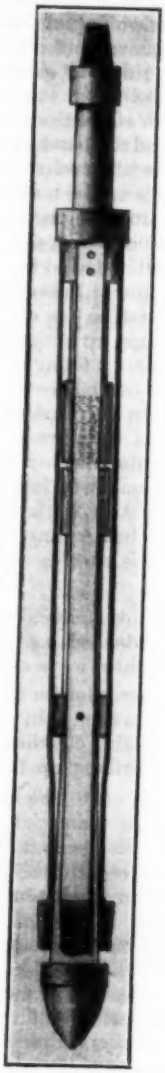


FIG. 10.

7 to 11 mm. thick. To do this, the casing shoe is first filled with stone, then the casing column is lifted some thirty feet or more, until a window is left between it and the lost shoe. (The casing must, of course, be pulled and a new shoe screwed on before the work is restarted.) A hoof bit is then used of as large a diameter and as eccentric as possible. Drilling should not be forced at the commencement, as subsequent progress depends entirely on whether a proper start has been made. It is essential that the bit should work evenly, otherwise a thin strip of casing will be left behind which will give no end of trouble and retard the work. At the commencement the hoof bit should be kept as flat as possible, *i.e.*, it should be dressed at an angle of 5 degrees to 8 degrees; as work progresses, the angle is increased to about 30 degrees. A suction sand-pump will be found to be the best means of keeping the bottom of the borehole free of cuttings. If a few inches of these are permitted to accumulate, they will prevent progress altogether.

Lost Casing.—Casing lost through a joint nipple pulling out can easily be recovered, either by using a die nipple or by taking hold of it with a casing spear. This depends, however, on the type of casing being used.

Runaway Casing.—Two or three thousand feet of casing may run away a considerable distance without it suffering practically any damage, and its recovery proves quite a simple operation, whereas a shorter string of casing may fall only a few feet, yet become so deformed, torn up, and telescoped that it takes many months of fishing to clear the well. When casing has run away, and it has been ascertained that considerable damage has been sustained, careful measurements must be taken, and only after a thorough study of the prevailing conditions should a grappling tool be lowered in. A casing spear can be used if the casing is not split, but it is only advisable to use that type where the hold can be released as desired. If the casing is split, then it is better to use a casing socket, which is passed over the top piece and a grip taken on the outside circumference; or a combination tool can be used which will take a grip both on the inside and outside. When strips of casing present themselves, an ordinary tongue socket can be used with advantage. In Galicia, fishing poles of from 1 9-16ths. in. to 3 in. diameter are used for this kind of fishing, and are made with either right-hand or left-hand joints, depending on the work to be accomplished.

Frozen Casing.—When casing has become frozen or seized, it can sometimes be freed by releasing the casing dogs and allowing the casing to rest partly on its own weight, held only by the blocks and tackle, then lifting and again releasing. One must be careful not to allow too much slack, or the casing might be deformed. If

this method is not successful, and it cannot be freed by jarring, tension fishing, or the steam method (see paper on "The Galician-Canadian System of Pole Tool Drilling,"* under "Fishing Instruments"), it may still be partially recovered by the following means:—

(*a*) By slitting the casing joint just above where it is known to have become frozen; the remainder of the column is then free and can be withdrawn. In some cases several joints have to be slit before a point is reached where the casing is free. If it is definitely known that only the casing shoe is frozen, it can often be freed by slitting it in two or three places throughout its entire length, starting just below the joint. The slits should be from $\frac{3}{8}$ in. to $\frac{1}{2}$ in. wide, and the casing-shoe being then no longer rigid, can be pulled loose.

(*b*) When the whole length of casing cannot be recovered, and it is considered expedient that drilling should be continued with the same dimension, one can either sidetrack the lost part or drill it up. Whichever method is chosen depends more or less on the formation. If there is much hard rock it is better to drill it up, but if the walls of the borehole are moderately soft, one would be quite safe in sidetracking. The difficulty is in getting a proper start. Once this is made, it is surprising how quickly the work proceeds. For instance, 6½ in. diameter casing of 9-32nd in. thickness can be collapsed and sidetracked at the rate of from 5 ft. to 10 ft. daily at a depth of, say, 4500 ft., and the working column will follow on without much difficulty. The proper tool for this work is a specially shaped hoof bit, and it is advisable to use a heavy sinker or even two coupled together. If the bit is properly dressed, not only will the casing be collapsed and sidetracked, but no underreaming will be necessary to enable the working column to follow on easily. The hole should be well sand-pumped out after every run, and a filling of stone put in, so that the bit has to work out the distance twice over. This goes quickly, and is not time lost but time gained, as one is quite certain that each foot has been properly worked out.

Complications Caused by Runaway Tools, Poles, and Casing.

Where tools, poles, and casing have run away, the operator must devise such methods and means as will suit this particular job, and no hard-and-fast rules can be laid down. Where tools and casing have run away, it is important to close the window between

* *Journ. Inst. Petr. Techn.*, viii., No. 34, pp. 617-665. Dec., 1922.

the two columns as quickly as possible, so as to avoid an accumulation of cavings forming on the top of the lost tools lying inside the runaway casing. The next thing to do is to run a swedge to make quite sure that the two casings are parallel with one another, and that the tools can be passed through them. An impression block is the next instrument to use, and if a number of poles have also run away, then it is better not to jar and pull, but rather to unscrew the poles one by one until the lost string of tools is reached, and these can then be fished out.

During fishing for the lost tools, it will have become apparent whether the casing has become bent or not; if it is found that the casing is only slightly deformed, or only to such an extent that it can be pulled through the preceding column, then a casing spear can be lowered in and a hold taken, after having controlled the casing with a swedge. It may be, however, that the runaway casing has become frozen meanwhile; to test this, the upper part of the parted column can be lifted three or four feet clear of the runaway column and then a pull taken on the rods. If it is found to be free, then both columns, *i.e.*, both the free and the runaway sections, can be pulled together.

One is sometimes confronted with tools having become wedged in the casing either during letting in or pulling out. When this happens a little upward and downward jarring may loosen them, but under no circumstances should the situation be forced either way, as the casing is liable to become damaged. The best thing to do is to endeavour to pull the casing with the wedged tools together. Should the tools have become stuck in a frozen column, one should try to unscrew the casing at a point below where the tools are wedged, and to pull casing and tools together, as previously mentioned. Should this not prove possible, owing to the casing being frozen above the point where the tools are stuck, the tools must either be wrenched loose or cut to pieces with a rotary cutter, the latter being the best and safest method.

Another frequent casing trouble is that known as "collapsed" casing, and it depends on the extent of the damage sustained as to the type of swedge it is advisable to use. If the casing is badly collapsed, it is better to use several swedges of progressive diameter, the plain fluted type being the best with which to commence operations. Once the casing is swedged back into something like its original shape and diameter, the roller type of swedge, Fig. 12, should be exclusively used, as this is not so liable to jamb and does not abrase the casing to the extent of the ordinary swedge. The work should never be forced; if the swedge is driven through with undue haste, the casing is liable to collapse above it, making its recovery a difficult matter.

Recovery of small objects, such as underreamer dogs, etc., which are difficult to remove by the usual type of tools, can in most cases be accomplished by putting clay into the hole and mixing this up

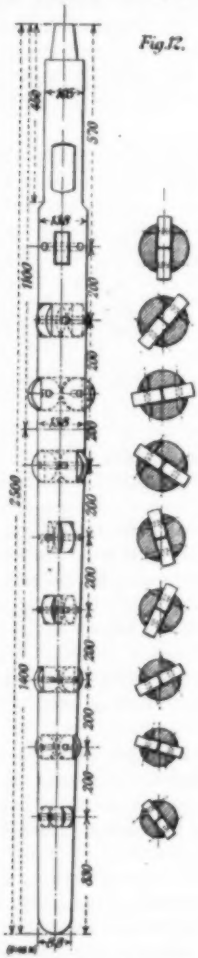


Fig. 12.

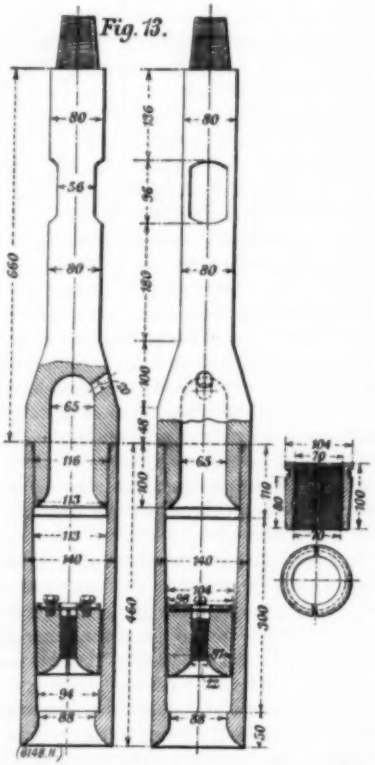


Fig. 13.

with the bit. A suction sand-pump is then lowered in, and this will generally remove the clay together with the lost bodies. Another means of recovery is to use a basket grab.

An Instance of Complicated Fishing with Pole Tools Arising from a Series of Mishaps.

The well in question was 4040 ft. deep with 6 in. movable casing hanging free from the casing dogs, the casing-shoe being 33 ft. from the bottom.

Whilst withdrawing the tools, the forty-fifth pole above the drilling jars broke, and the tools together with the 45 poles ran away about 680 ft. On the tools striking bottom, the third pole above the drilling bit broke and 42 poles ran down alongside the tools. Of these 42 poles the twentieth broke and ran down alongside the others, wedging them tightly. All the poles were extremely bent and cork-screwed.

After an impression was taken, a tongue socket was lowered in and a hold taken on the length containing 22 poles, considerable upward jarring resulting in the recovery of five poles, the fifth having broken off just above the lower joint. It was at this point that the first mistake was made. Instead of measuring the borehole after the first five poles were pulled out, a socket was let in without any precautionary measure being taken, and instead of gripping hold of that length where the five poles had broken off, a grip was taken of the length containing 20 poles and upward jarring was resorted to. The longer they jarred, the more tightly the poles became wedged, with the result that the socket was left behind with jars, sinker and a further number of poles. A second socket was let in and jarring continued until the top pole of the 20 broke off, and this, together with the two sockets, was withdrawn. The socket was then let in time after time, and after several days' fishing there still remained 30 poles and the lost tools in the hole, these poles being in three separate lengths, tightly wedged one alongside the other.

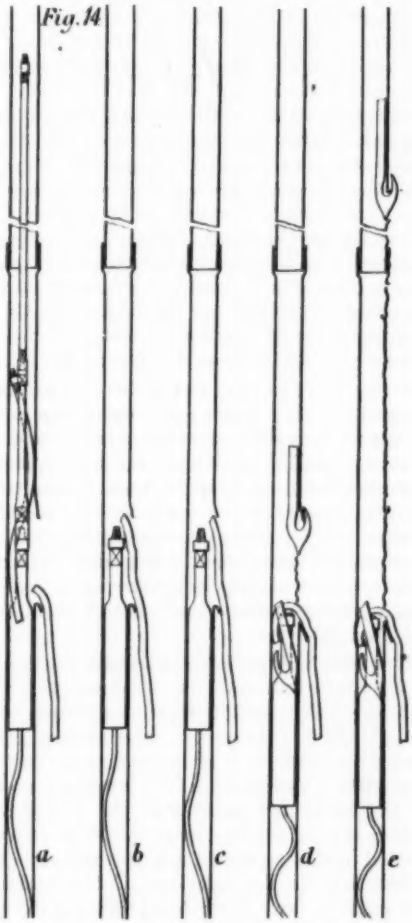
When it was found impossible to get the poles out by incessant pulling and jarring, those in charge of the operations began, for the first time, to reconsider the probable position in the borehole, too late, however, to form any definite idea of the exact position of the runaway poles and tools. There now remained only one course to pursue, *i.e.*, to unscrew the poles one by one, always taking hold of the one which presented itself uppermost. This took a good deal of time, and eventually there only remained the string of tools connected to two drilling poles, plus one and three-quarter poles which had run down alongside. At this juncture undue haste proved a false step, and was the commencement of one mishap following another. Instead of continuing unscrewing until only the tools remained, the casing was lifted to allow the one and three-quarter poles to fall through. A fishing socket, Fig. 13, was then let in and a hold taken of the two poles connected to the tools, pulling

was commenced, and, judging by the continued heavy pull which was required, it was evident the tools had become somewhat bent. During pulling, the poles broke and the whole thing ran back again, together with the fishing socket, plus two more drilling poles. Left-hand fishing poles of 1 9/16ths in. diameter were then lowered in with a heavy fishing socket, by means of which the two poles, together with the lost socket, were recovered; after this, hold was taken on the two drilling poles coupled to the drilling tools.

The work of pulling out went well until there remained only two left-hand poles (plus the lost tools and drilling poles) to be withdrawn from the borehole, when carelessness on the part of the driller's helper (due most likely to over-excitement) to properly screw on the pole swivel to the poles, resulted in the swivel and poles parting company when the next lift was taken, and the drilling tools with the two drilling poles, plus the socket and two left-handed fishing poles, ran away to the bottom. An impression block was now run which showed the pin end of a fishing pole standing vertically in the centre of the borehole (*a*, Fig. 14). Heavy left-hand fishing poles were again let in, and a connection was made by screwing on; but, after considerable jarring and pulling, it was found that the tools would not budge. The poles were then uncoupled, and a die socket let in to unscrew the two left-hand poles which had run down. After making a connection and unscrewing, the die socket was withdrawn, but it brought only one left-handed fishing pole with it to the surface, leaving the second fishing pole in the borehole. The die socket was again let in, but no connection could be effected, therefore it was withdrawn. On an impression block being run in, it was found that instead of showing, as was expected, the pin of the second fishing pole standing vertically, the impression showed the fishing pole lying over to one side. An ordinary tongue socket was then let in which resulted in a piece of fishing pole some 6 ft. long being withdrawn from the borehole. The next impression obtained showed the substitute's pin, and the substitute was then unscrewed from the socket and pulled out. Further impressions showed the pin of the lost fishing socket, but no trace of the broken fishing pole. As the body of this socket was only $\frac{1}{4}$ in. smaller than the inside diameter of the casing, it was difficult to know where the six metres of fishing pole had got to, but by running a deeper impression block which would pass down at least to the shoulder of the socket, it was ascertained that a quantity of sand had collected on the top of it. A small indentation on one side of the impression block immediately gave the clue as to what had happened. The missing part of the fishing pole had been driven completely through the wall of the casing, and only the bent and broken end was protruding about $\frac{3}{4}$ in. inside. After

using various hooks and spuds, the broken-off pole was pulled out about 1 ft. 8 in. into the casing (*b*, Fig. 14), but owing to the angle at which it was protruding, it was impossible to get hold of it.

Fig. 14



The pole was then driven back until the broken end became practically flush with the wall of the casing (*c*, Fig. 14).

A connection was made by screwing directly on to the fishing

socket and upward jarring was commenced, which proved to be a very false step. After some fourteen days' continued upward jarring, the pin was broken off the socket, and no further headway could be made. It was then decided to try to drive the socket downwards, but instead of first making a connection on to it and driving from above by means of a pair of fishing jars, a driving bar was made, having an outside diameter practically equal to that of the inside diameter of the casing; this was lowered in and driving down was commenced. The depth at which the driving bar stood was not measured. After several days of heavy driving, which only resulted in the tools being driven some 2 ft. 6 in., the driving bar was withdrawn. An impression block was run in which showed an impression of the fishing pole bent in the shape of a hook and lying right across the borehole and over the top of the socket stem. What had happened was that, instead of driving on the top of the socket as was intended, the driving bar, owing to its fullness, had come to rest during lowering in on the broken fishing pole which was protruding slightly inside the casing, and on this they had been driving until it had become bent over in the manner described.

Under these circumstances, it was quite impossible to effect a connection with the fishing socket, and there was only one thing that could be done: to make a circular cutter to cut off the bent pole flush with the casing, and after having accomplished this, the severed portion could easily have been recovered, enabling a connection with the socket to be made again. Instead of this, a hook was constructed by which it was anticipated it would be possible either to withdraw the broken pole altogether or to straighten it out. The hook was duly lowered in, and after several hours of upward jarring it broke, and about 2 ft. of this hook was left behind (*d*, Fig. 14).

This, however, did not prove a sufficient lesson, so a second hook was let in, which took hold of what was thought to be the bent fishing pole. After several days of upward jarring, during which time about 33 ft. of progress was recorded, the hook was uncoupled and withdrawn, and after running an impression block it was discovered that instead of a hold having been taken on the broken pole, the hook had penetrated the casing at that point where the pole had gone through, and the 33 ft. of upward jarring had only resulted in slitting the casing to that extent (*e*, Fig. 14). It was now that a further serious error of judgment was made, for instead of cutting away the pole and freeing the socket, it was decided to pull the casing, poles and tools together, the result being that the casing parted at a joint which had been slit.

An impression block was run in; became trapped, and could not be recovered. During this process a half pair of fishing jars was

also left behind. The decision was then come to that there was nothing more to be done, and the best course would be to drill alongside, or, in other words, to sidetrack some 136 ft. of 6 in. diameter casing containing a bit, sinker and jars, two drilling poles, a fishing socket, two feet of hook and impression block, and a half pair of jars, as well as the cause of the trouble—the broken fishing pole. The work of drilling past took over seventeen months to perform.

By this instance it will be seen how very easy it is for a simple fishing job to be converted into a serious fishing complication through lack of spending a little more time and concentrated thought at the outset, and by taking risks through undue haste to get the well cleared.

Had accurate measurements been taken in the first place, it would have been ascertained that the poles had broken in two places, and had run down alongside, thus becoming bent and wedged. Then, instead of pulling and jarring, the hole could soon have been cleared by the simple process of unscrewing whatever the socket took hold of. This would certainly have taken less time, and would have been the safest course to pursue.

After the impression block had been run and the borehole measured, a hold could have been taken with a suitable socket, and a little upward jarring tried, which would have tended, not only to straighten the bent poles, but also to loosen their joints. Unscrewing could then have proceeded until all the drilling poles had been withdrawn and only the lost string of tools remained, which could have been caught hold of and pulled out without much trouble.

During this fishing scrape the following errors of judgment are apparent, and were the cause of twenty-one months costly and tedious fishing.

1. *In not measuring the well* after the removal of the first five rods.

2. *In lifting the casing.* This is always a step which should be given most careful consideration during fishing operations, as there is always the possibility of the walls of the borehole caving, or that the tools may fall off to the side, rendering their extraction a more complicated matter. Should the casing be lowered back again, there is every likelihood of the lost object being driven to one side and the casing becoming damaged.

3. *The continued upward jarring* after the broken fishing pole had been driven flush with the casing, and a direct communication was made on to the socket. It was obvious that in a case like this upward jarring would be of no use whatever, and could have no actual loosening effect, because underneath the socket were two drilling poles which were more or less bent and cork screwed, and

which would serve to act as a spring; below the poles were the drilling tools which had become stuck, thereby acting as an anchor. It should have been evident that only one of two things could happen—either the drilling poles above the jarring bar would break, or else the pin would be jarred off the socket, as was the case (see "Jarring," "Galician-Canadian System of Pole Tool Drilling").*

The best thing to have done in this instance would have been to let in 2¾ in. fishing poles, a firm hold taken, then the tools could have been pulled together with the lost fishing socket by means of ordinary tension fishing. The end of the broken fishing pole which was protruding slightly in the inside of the casing would have been pushed flush with the walls of the casing, thus affording a free passage for the tools.

4. *Driving the socket down.* In the first place, the idea of putting in a loose driving bar was simply asking for trouble; and, secondly, taking into consideration the fact that below the socket were two drilling poles (total length, 72 ft.) which would act as a spring, what progress could be reasonably expected?

However, since it was decided to drive down, instead of using a loose driving bar, a connection should have been made with a taper die socket on to the implements it was intended to drive down; above this would be the fishing jars, followed by the driving bar. By this method there could have been no doubt of achieving the purpose aimed for.

5. *When it was discovered that, instead of driving the socket downward,* they had been simply bending the broken fishing pole over the stem of the socket, there was clearly only one course left open: to cut off that part of the pole protruding into the casing flush with the casing wall. The lowering in of a hook was a great mistake, especially as it was known that the casing had already been holed. The only kind of hook which could be used with any degree of safety in such a case would be of similar construction to the "bootjack."

6. Probably the most serious mistake of all *was the decision to pull casing and lost equipment simultaneously,* after it was definitely known that the casing had been slit to the extent of 33 ft., and that a joint had been cut through. The very fact of the 6 in. casing having in all probability become deformed by the penetration of the fishing pole, and the possibility of difficulty being experienced in pulling the deformed 6 in. string through the 7 in. casing column, should have caused the utmost care and forethought being given as to the advisability of such an operation, quite apart from the fact that it was known that the 7 in. casing was deformed, a

* *Journ. Inst. Petr. Techn.*, viii., No. 34, pp. 617-665. Dec., 1922.

number of meters having collapsed during drilling operations, and which had had to be swedged out. The parting of the casing at this juncture was in all probability the best thing which could have happened in the interest of the well in question.

In a short paper it is not possible to give particulars of all fishing implements (see "Fishing Instruments," "Galician-Canadian System of Pole Tool Drilling")* as specially constructed tools have to be made to suit different conditions. Although this paper deals principally with pole tool fishing, many of the instances given are applicable to other drilling systems.

In ordinary accidents, standard tools are used, but specially devised and constructed tools are in all probability never used a second time, or are converted into a different type of instrument altogether, thus ceasing to exist.

It has been the writer's endeavour to set out a few everyday fishing jobs and remedial methods adopted. There are, however, so many combinations possible in practice that a paper many times this length would not suffice to deal with them all in detail.

DISCUSSION.

The President, in opening the discussion, thought the author had made it clear that more haste was often responsible for less speed, and that considerable time should be spent in thoroughly mastering the situation before any work was done. The author had referred to the desirability of imagination being displayed in connection with the work that had to be carried out, and it meant that a man must be employed with special training and with a special personality, because it was impossible to teach a man all the little things which were so important in the carrying out of work which called for ingenuity and resource at every moment of the operation, otherwise, as the author had stated, very great difficulties might arise.

Mr. A. Beeby Thompson said that in his professional capacity he was constantly called upon to specify fishing tools for outfits going to different parts of the world, but he found that whatever he specified it was always criticised, and the statement was made that he never sent the things which were required. If the catalogues of all the supply houses were searched, and every tool connected with such operations was sent out, they would still fail to provide for all eventualities. Personally, he considered all depended on the resourcefulness and skill of the man on the spot. Difficult fishing operations could only be performed satisfactorily by men of suitable temperament. As the author had stated, imagination was an

essential qualification; they required the capacity of being able to picture what was transpiring or had occurred to tools working at a depth of several thousand feet, linked merely by a light cable or a thin string of rods. The more educated type of driller made sketches and drawings of what he thought had taken place when the accident occurred, but the ordinary driller did not do so. Nevertheless it was perfectly remarkable how, notwithstanding the entire absence of theoretical knowledge, drillers were able to know instinctively what had happened, and select appliances which would get them out of their trouble. There were several main types of fishing tools. In Europe a type was used which generally had some positive connection with the surface, but in America percussion drillers still clung to the cable and bull-dog type. In America fishing poles were unknown, but on the Continent of Europe no company drilling on any scale was without a string of fishing poles or tubes. In the old Russian fields the drillers would never, under any circumstances, lower a fishing tool except with rods which gave them absolute control, either to connect or release them at will. When unable to avoid the use of a positive connection with lost objects, a string of casing was always used. The American practice had been varied somewhat by the introduction of the rotary, because, naturally, with rotary drilling the drill-pipe constituted a positive connection with the fishing tools. In the next place he desired to emphasise the really remarkable advances in sidetracking. Until recent years sidetracking on the scale now attempted was impossible, but owing to the enormous strength of present-day tools, the great power available by the larger engines, and the very high-grade metal which manufacturers were using, it was possible to tackle almost any difficulty. No matter how serious the accident, it was now possible to sidetrack the obstruction by means of heavy bits. It might be argued that if that were done crooked holes resulted. That was no doubt the case, but it did not seem to affect the situation very much, as the slight deviation occasioned appeared to little influence the speed of drilling or the freedom of casing. He knew of cases within the last few years where the drillers had been able to sidetrack 300 or 400 feet of casing in about a week, a really extraordinary performance. That many wells were crooked without the knowledge of the owners was quite certain. He could recall a case which occurred quite recently in Burma, where the pump tubes in a well of a company with which he was connected started dancing in the well, to the consternation of the field manager, who, however, discovered that the movements of the tubing synchronised with the oscillations of the walking beam of a neighbour's drilling well. Equally mystified was the driller of the adjoining well by the action of his tools. It then

became apparent that the new well was drilling into the old producer. It was subsequently ascertained that the old well was at fault, and had been producing for years from the neighbouring property. On investigation it was found that the producing well was deflected about 45 feet between 700 feet and 1500 feet, showing how great a deviation could occur without the owners being acquainted with the fact. Drillers were not always able to say when an accident took place, because it was not always easy, especially in rotary drilling, to detect a twist-off. Drilling operations might go on for some time before it was known that an accident had occurred, and will then become badly damaged or jammed. He was quite sure that many fishing scrapes resulted from curvature of hole, and many difficulties were attributable to the same cause, in spite of opinions to the contrary on the part of many drillers. It was sometimes said that the tools would not work freely in a crooked hole, but, as a matter of fact, they often did. In one case recently which came under his notice the drillers on a fairly deep rotary hole had endeavoured in vain for a very long time to get the casing down. They repeatedly reamed under the belief that it was due to cavings, but still casing would not go down. He expressed the opinion that it was a crooked hole, and that if means were taken to straighten it the casing would pass freely. As a result they attached to the top of the bit about 40 feet of casing and then rotated this to bottom. After that the casing was put in, and it passed freely to the bottom without any obstruction, showing that curvatures or kinks in the hole had been the cause of the trouble; the unguided bit on the rotary drill pipe merely following the previous passage.

Mr. Ashley Carter said he desired to draw attention to one very important point in connection with the prevention of fishing troubles. It was almost universal to find the derrick, the tools, and everything else connected with the drilling in a disgracefully dirty and untidy condition. Cleanliness on the rig stimulated the interest of the driller; it revealed defects in the tools; it cost nothing, and was a means towards the prevention of fishing troubles when the various operations were undertaken. Cleanliness, which was so much neglected at the present time, was so easy to attain that he had thought it worth while specially to draw attention to the point.

Mr. Gordon Pitt said that he did not suppose there could be many present who had actual experience of such seriously complicated fishing operations with Galician pole tools as some which the author had described, but probably some of them had had to undertake jobs of that kind and could therefore very sincerely

appreciate the advice the author had given. Although such a paper was difficult to discuss, it was none the less a paper which would be of great use to a considerable number of members present, and would, he thought, probably be of more use still to a great number of the absent members who would eventually read the paper in the JOURNAL. He did not desire to criticise or discuss the paper in any way, but only to throw out the hint that if any members possessed any practical experience on the subject, particulars of which they could give in the course of the discussion, it would prove most useful to the members in the more remote parts of the world who would eventually receive that information through the medium of the JOURNAL. One never knew but that one particular piece of information might become available just at the very moment when a member on the field might want it.

Mr. F. G. Rappoport said he desired to ask the author a few questions. In the first place he would like to know if grabs were used at all in the Galician system. In the Russian fields the grab had been found a most invaluable instrument, which was almost human in its application. With reference to the drawing the author had shown of particular fishing rods, he did not quite understand how either a right-hand rod or a left-hand rod could be used singly. In the Russian fields the most useful fishing rods (either 2 in., 2½ in. or 3 in.) were those which could be used either way, by means of a simple locking arrangement. Perhaps the most interesting part of the paper was that it brought home very forcibly the disadvantages of the pole-tool system of drilling, especially at great depths, and brought into contrast the respective merits of the old and new systems of drilling. Up to a certain point he quite understood that pole-tool drilling was applicable, and in Galicia he had no doubt that the drillers had become so accustomed to it that the change to any other system was difficult. When it was only necessary to deal with caving ground at shallow depths, he had no doubt that a system like the Russian system or the Galician pole system was perfectly applicable, but at great depths, together with caving and bad ground, it made one begin to think very hard when one saw a complicated fishing job such as the one described in the latter half of the present paper, caused entirely by broken rods. It was a question whether, after all, under such circumstances the old system had not come to its end, and the new system be rung in. Many members like himself had great affection for the pole-tool system; they had been brought up on it, but one's respect for it was now tinged with a good deal of veneration.

The lesson that had been brought home to his mind after listening to the paper was whether the pole-tool system should not give way to some more modern system, particularly at greater depths.

The President said that, before calling upon the Secretary to read certain communications which had been received, he would like to ask one or two questions. The power of the lift which was usually put on must, of course, vary under certain conditions, but it had occurred to him that with a long string of rods the weight alone must very greatly strain the upper rods. If a jam occurred lower down it seemed to him that they were getting somewhere near the limit of the strength of the metal. As Mr. Rappoport had suggested, it appeared to him that the poles were a source of danger, because if they broke they were very difficult to remove. A very little jamming action at such a depth would, he thought, produce a stress equal to the tensile strength of the material.

The Secretary read a written communication from **Mr. Alfred W. Nash,** which stated that to the oil man without practical experience of field work the author's paper would undoubtedly give the impression of being very empirical. It must be borne in mind, however, that fishing methods could not be learned from a book, as no two fishing jobs presented the same difficulties.

Many oilfield men did not realise the importance of the application of engineering principles to fishing methods; the author was fortunate in having gained many years of experience on a field where every advantage was taken of engineering skill and knowledge.

The average driller was judged by the speed with which he "made hole," but that part of a driller's education was by far the simplest.

The qualifications necessary to the making of a good driller should be in the following order of importance :—

(*a*) Skill in keeping out of fishing troubles generally.
(*b*) Skill in carrying out fishing jobs successfully when they did occur.
(*c*) Skill in drilling.

For success in (*a*) and (*b*), experience was not the most important feature, as was proved by the fact that many oil men of lifelong experience, although splendid drillers, showed up very poorly in a fishing scrape.

The most important qualification was the ability to see in the mind's eye what had occurred, how it had occurred and why; to invent and design tools with a due regard to proportion and with a firm previous conviction of how they were going to work—in other words, the knowledge of the engineer. Once invented, the experience of the driller was necessary for the intelligent use of such tools.

The cause of most fishing scrapes was not through lack of attention in what was going on down below, but rather from that which the driller had omitted to do at the surface.

Under the heading "Lost Tools" mention was made of a horn socket having been used after the top of the bit had been rasped away. As it was necessary for a bit to stand up fairly firmly in the hole in order that rasping might be done successfully, it would be interesting to know whether Mr. Miller would not agree that a slip socket would give a much more positive hold than a horn socket, as the latter depended on a friction hold entirely.

Also under the heading, "Stuck or Lodged Tools," a little clearer description of how the tongue of a socket could enter the hole of a rope socket when the cut-off portion of the drill line still remained in that hole would be of interest. Would it not be better, before cutting the drill line, to pass a bumper over it and try bumping the tools downwards?

It would also be of interest to hear why the author preferred slitting the casing joint in preference to cutting the wall of the casing horizontally. Slitting a joint at great depth would appear rather risky, and by lowering a trip spear on a set of jars, taking hold, and jarring upwards, it was a simple matter to find out whereabouts the casing was frozen and where free and cut accordingly.

The underreamer dogs mentioned must be very small to allow of their being drawn into the body of the sand pump. Even assuming 6-inch casing was going down, the pump barrel could not be much larger than 5 inches, and, allowing for increased thickness at the bottom, the entrance to the same pump could not be bigger than, say, 4 inches diameter. Would the author state whether electric magnets had been used in Poland for the recovery of small objects?

One point had not been mentioned in the paper, which was a very important one—viz., the examination of drilling and baling lines. Some drillers examined their drilling lines fairly frequently, others only examined them after they had parted, but it was surprising how very few paid much attention to the sand line. That was partly due to the fact that the recovery of a sand pump or bailer did not usually present much difficulty, but such fishing jobs meant a loss of time and money and might in a caving formation lead to difficulties with the casing.

A communication from **Mr. E. K. Wallen** was then read, in which he entirely agreed with the author's views on the importance of keeping an exact record of measurements and numbers of everything lowered into a well. Where that was made a rule, and was carried out thoroughly, those in charge of the operations were in a position of having complete command of the situation if trouble occurred, as it generally did sooner or later.

Those measurement could, of course, be avoided to a great extent by standardising tool joints, squares, lengths of squares, collars, etc. He did that in Russia, making the tool joints in three

sizes—viz., large, medium and small. When that system was practised one could always obtain from the Materials Store an exact duplicate of the tool being fished for, and thus fit the fishing tool accordingly.

In fishing for a lost bit he had always found it a good policy to make sure that the bit was free before any tool was lowered to take hold of it. It was surprising how a lost bit would stick in soft formation, although apparently it had only been hit a few times with the sinker bar, and the operators were almost sure to lose a few socket-dogs and ends of hooks before coming to the conclusion that they must spud it loose or drill by it, as the case might be. Then, having got those pieces of steel and iron into the hole, one had to contend with the further trouble which their presence was bound to cause by greatly interfering with the spudding or drilling. A spud was the proper tool in a small hole, but in a large hole he always used a bit a few inches smaller than the one lost, and drilled away as if nothing had happened until the lost bit was completely freed ; then a light hook would do the rest.

With regard to the author's suggestion that it was advisable to remove all water from the bore hole, his experience had been that very slow progress was made with oil only in the hole. Clay did not mix well with oil, and the cuttings hung close to the bottom and did not rise, as was the case with water. A good way to drill through sticky clay was to use a lighter sinker bar, and also put a light one on top of the jars, run loose, and use the jars. In cases where the sticking was not so bad, they should just run loose, and the drilling would go ahead. That was, of course, a very rough way of drilling, but it certainly paid, and in his experience was about the only instance when it paid to be rough in drilling. What happened was that, running loose, the tools did not get the full benefit of the beam stroke, consequently the blow was not of full force ; furthermore, on the up stroke, they were jarred loose if the bit happened to be stuck in the formation. The so-called " sticky-clay " in most cases was shale, free from sand or other grit, and after being broken up by the bit it became a puggy, sticky substance at the bottom of the well. He had little doubt that it was in that that the tools stuck, and not in the actual formation in its original state. A few strands of wire rope thrown into the hole were very good when the sticking was really bad. They should be cut a little shorter than the gauge size of the bit.

With regard to frozen casing, he thought the first thing to do was to try to press the casing down with a screw, or hydraulic jack. If then it did not go down, the next best thing to do was to leave it there, and put in the next column. Very often, however, the casing

must be loosened, in which case the measures to be taken would depend entirely upon the cause of the freezing.

Mr. Henry F. Moon, in a written communication, raised two points, firstly in regard to the paragraph in the paper dealing with slightly bent tools, and, secondly, the paragraph dealing with sidetracking. He thought it must be very dangerous to try and straighten bent tools by jarring them through the casing shoe and risk getting the shoe deformed or parted, so that it could not pass through the next size casing, as then it would be a very difficult, if not impossible, operation to pull the bent tools and casing together. He should certainly prefer to start stripping the moment he discovered the tools were bent sufficiently to prevent their passing the shoe, and so avoid the possibility of having to sidetrack some of the casing and shoe as well as the lost tools. It seemed probable that, knowing the conditions of the formation and the danger of losing a second string by drilling past in the usual way, time would be saved in the long run by using a long starting bar at the outset, when it was found impossible to free the original string, as should two or more strings be lost side by side in a six-inch hole, it would possibly be very difficult to keep the hole straight in drilling past even with a long starting bar as described.

Mr. Albert Millar, in reply to the President's question regarding the upper rods of a long string of drilling poles being strained, and to the weights of lifts taken on fishing poles, said that conditions varied considerably during drilling, and "X," the unknown quantity, played a very important part, as for instance :—

(*a*) The friction on the poles in casing where the borehole is crooked.
(*b*) The resistance offered to the free lifting of the drilling bit, caused by caving, when drilling in "Gumbo," or when the hole is not properly sandpumped out, and the drill is running in heavy sludge (detritus).
(*c*) The magnetic pull of the casing.

An instance of maximum stress under ordinary drilling conditions can be taken :—

The stress on the uppermost rod of a string of poles working in a borehole, 1350 metres deep, with a heavy set of 7 in. tools, and making allowance for the unknown quantity "X," would be approximately from 6000 to 6500 kilogrammes.

The drilling poles are generally of 22 millimetres diameter, and are made of best quality iron, having a tensile strength of from 36 to 42 kilogrammes per square millimetre, the percentage of elongation being from 20 to 25 per cent.

The chief causes of drilling poles breaking are :—

Careless or inefficient drillers not running their tools properly, drilling in "Gumbo," running poles which, through long or misusage, have become crystallized or strained, through insufficient care being taken when welding on pins and boxes. This latter is perhaps the most frequent cause. Also, badly worn joints often result in drilling pole trouble. If sufficient care is exercised in selecting the right material, and in welding on the pins and boxes, regularly examining the joints, replacing those rods which have become strained and avoiding mishandling during drilling, there is no reason why poles should be inferior to cables as regards durability.

In tension fishing, lifts up to 150 tons, and even sometimes as high as 200 tons, are taken, and it is not unusual to hear of fishing poles being torn apart. As an instance of a long and big pull, the following is a good case in point :—A string of 3 in. diameter steel fishing poles, 1200 metres in length, was stretched somewhat over 11 metres in a period of 2½ months continuous tension fishing. The poles were pulled from 10 to 15 centimetres per lift by means of "fishing jackscrews," and then given from three to twelve hours, and sometimes longer, to recover. The individual length of each pole before the commencement of fishing operations was eight metres ; after being withdrawn from the borehole, it was found that some of them had been stretched from 30 to 70 centimetres.

The poles nearest the top of the borehole suffered most, and a number of them were no longer of any value. This particular string of fishing poles weighed over 38 tons.

Fishing poles are generally made from a steel having a tensile strength of from 40 to 48 kilogrammes per square millimetre, the percentage of contraction being 48 to 56 and of elongation 36 to 41.

He had been trying for some time past to obtain reliable figures with regard to the weights and lifts taken during fishing, and also in regard to the amount of power which was transmitted from the top to the bottom of the borehole, or in other words, the percentage of total energy expended that was brought to bear on the object that had to be fished out. He did not consider the figures he had already obtained were sufficiently conclusive or reliable to be made public, but he hoped at some future date to be able to give some reliable statistics. He had stated in his paper that he considered jarring was the best method of loosening objects which had become stuck in the borehole, not by means of a jarring that was half pull and half jar, but by submitting the object that it was desired to recover to a number of sharp upward blows in very quick succession. Jarring of that nature set up vibrations in the lost object and helped towards its release. Should that method not be successful

tension fishing could then be tried, but he advised that the latter method should only be used as a last resource. That could not, however, be laid down as a hard and fast rule, because each fishing job must be studied on its own merits. In answer to the questions asked by Mr. Rappoport, he desired to say that grabs such as he mentioned were not used in Galicia, or at least not generally used. All strings of fishing poles were made either right or left-handed. Left-handed poles mostly were used when fishing for tools, and right-handed ones when fishing for casing. New systems so far as Galicia was concerned were coming into use, in fact several rotary and cable outfits were already at work. In answer to Mr. Nash's queries he desired to say that the horn socket referred to in the paper was a split socket fitted with a tongue. If it was necessary to file a head off the lost bit it must first be straightened up and tamped so that it did not fall over to the side again. In reply to the question as to how the tongue of a tongue socket could enter the hole of a rope socket, he desired to say that the socket referred to was an over-shot socket designed to take a grip on the outside. The horizontal type of casing cutter was in some cases very much better than the slitter. With Canadian tools, however, the casing slitter was mostly used, and he had never heard of an accident happening owing to its use. He had also never heard of electric magnets being used in Galicia, and he was not certain that they had been a success elsewhere. He quite agreed with Mr. Moon that to try and straighten out bent tools by jarring through the casing shoe was a dangerous practice. Sometimes, however, the tools were only slightly bent, and, after a little jarring, could be made to pass inside the casing shoe. It was all a matter of judgment, and if it was found that considerable jarring was necessary, other remedies should be tried, such as stripping or sidetracking. If only one set of tools was left behind in the bore hole, and it was decided to drill past, a starting bar would hardly be necessary unless the wall of the borehole was extremely hard, preventing driving to the side.

The President said he estimated from the author's remarks that the weight would be 29 tons to the square inch, which was pretty close to the limit. He was sure the members had been very deeply interested in the paper, which was of a most practical nature, as it illustrated the operations that had to be performed, and called attention to the many difficulties involved, and that they would desire to join with him in passing a very hearty vote of thanks to the author for it.

The resolution of thanks was carried by acclamation, and the meeting terminated.

Contributions from the Chemical Laboratory of Case School of Applied Science, on the Lubricant and Asphaltic Hydrocarbons in Petroleum.*

By Charles F. Mabery.

Although much time has been devoted in this laboratory to the composition of the distillable hydrocarbons in petroleum, no attention has hitherto been given here, and little elsewhere, to the identification of the hydrocarbons that cannot be distilled without decomposition. Of the few attempts to separate these constituents of petroleum by cold solution and precipitation, "cold fractionation," the most noteworthy is the work of Charitschoff, who described the following hydrocarbons, with their specific gravity: $C_{19}H_{36}$, 0·8930; $C_{21}H_{40}$, 0·9050; $C_{22}H_{42}$, 0·9080; $C_{24}H_{46}$, 0·9130; $C_{25}H_{48}$, 0·9150; and it has doubtless suggested the frequent allusions to the presence of naphthene lubricants in American petroleum. But none of the hydrocarbons from Baku oil, described in this paper, contain the series C_nH_{2n}, although some of these specific gravities are about the same as those of the series C_nH_{2n-6} in Baku oil to be described later, and none of the varieties of American petroleum have shown such composition.

Since petroleum hydrocarbons begin to decompose in distillation at about 200° C., and above 300°, most crude oils, even under pressures reduced to 20 mm., show evidence of decomposition, it is impossible to separate by any form of distillation the constituents of petroleum that will not distil at 300° in a vacuum.

Separation by Fractional Solution.—With the exclusion of distillation the only remaining possibility appeared to be fractional solution, and in view of the variations in other physical constants, there seemed to be no reason why the different series and homologues should not possess sufficient differences in solubility to permit of their approximate separation in this manner. Trial of the various solvents excluded all but a mixture of ether and ethyl alcohol, and since all the constituents of petroleum dissolve freely in ether, but are quite insoluble in alcohol, it seemed possible to prepare from them a convenient solvent. For general use a mixture of equal parts by volume, with suitable variations for the more soluble lighter ends, and the less soluble constituents of the heavier ends proved efficient for all the varieties of crude oil. For convenience

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of reference, the lighter fractions will be referred to as the higher or upper ends, and the heavier to the lower end of the series or group.

Under a pressure of 30 mms. the crude oil was first distilled to 300° C. For the removal of the paraffin hydrocarbons from the Appalachian oils, the solid residue was dissolved in ether to a dilute solution, alcohol added until the paraffin began to precipitate floculent, the solution cooled to 0°, filtered cold, again cooled to −20°, and again filtered, with very little paraffin remaining in the oil after the first filtration. There is some difficulty in reaching the point where floculent precipitation begins without carrying down a large amount of the semi-solidified oil.

Homologue Separation.—In lots of 1000 to 1500 grams, the vacuum residue, free from paraffin, was heated to the boiling point of the solvent in flat cork-stoppered bottles in a hot water bath, with frequent shaking, the stopper being held in with the finger and frequently removed to relieve excessive pressure. For collection of the homologues of all the series into ten or fifteen groups, the hot solution was poured off from each extraction, and the solvent distilled, the first fractions containing the more soluble upper end, and the diminishing solubility giving the consecutive fractions down to the last residue. For further separation of the series homologues, the lowest group was heated with the solvent of proper concentration, sufficient to dissolve a considerable part, and the hot solution poured off, cooled, and again poured off from the separated oil. This was added to the next fraction, which was again heated, the solution being poured off for the treatment of the next fraction. This procedure was continued to the upper end. (No PP.)

The solvent distilled off from the last treatment gave the first member of the group, and this procedure was repeated six times. Since the specific gravity of the fifth and sixth fractions was approximately the same, it was assumed that the homologues of all the series were fairly well collected within the respective groups. The efficiency of this method appeared in the differences in consistency between the lowest fractions, extremely thick viscous or nearly solid lubricants, as in the Appalachian oils, or thick black tarry or solid asphalts, as in the Texas and Russian oils, and the upper fractions, thin, amber-coloured lubricants.

Series Separation.—Beginning at the lower end of the group each fraction was about one-half dissolved in rich, hot solvent, decanted, leaving a residual oil, H-, the solvent distilled, giving another residual oil, D-, and this was continued with all the fractions to the upper end. To be sure that a single extraction gave an approximate separation, it was followed by another similar treatment,

giving two series, Da and Db. The specific gravity of a and b proved to be sufficiently concordant to indicate a fairly satisfactory separation by the first treatment.

In a second mode of series separation each of the first group fractions was heated to boiling with the solvent, the hot solution decanted, cooled, again poured off from the separated oil and distilled, giving three oils, H–, the first residue, C–, residue from cooling, and D–, residue from the distilled solvent. Each of these oils was again treated in a similar manner, and this continued three or four times, thus dividing the original oils into eight or twelve fractions, did not materially change the specific gravity from the results of a single cross separation, from which it was inferred that the marked difference in solubility gave a fairly good separation in the first extraction. The following examples of its application on some of the oils as a means of control show the rapid separation by this method. A top fraction of a Russian vacuum residue containing more soluble, heavier carboxyl constituents brought up from below, and another from the Rosenbury oil, were separated by a single extraction into light and heavier constituents as follows :—

Russian fraction, sp. gr. 0·9236 { 0·9594, H-residue, hot
0·9198, C-residue, solvent cooled
0·8925, D-residue, solvent distilled

Rosenbury (Penn.), sp. gr. 0·9064 { 0·9230, H.
0·8908, C
0·8875, D

No doubt the petroleum hydrocarbons under the condition of cold solution used by Charitschoff do exert a mutual solubility, and interfere with the use of specific gravity as a means of identification, but under the influence of a hot solvent it seems to be quite otherwise.

The constants relied upon for grouping and identification were : specific gravity, molecular weight and composition by analysis. Determinations of specific gravity were made in a Sprengel pyknometer at 20°, except of the most viscous tars, which were weighed under water by the method of Kirschbraun.

In the beginning, molecular weights gave much trouble, especially of the heavy hydrocarbons. Of the common solvents benzene alone at the boiling point was applicable, and this was reliable only with the lower members of the Appalachian oils. Stearic acid at 50° proved to be more satisfactory. A weight of oil from 0·3 to 1·5 grams, depending upon the specific gravity of the oil, gave a depression of from 0°·150 to 0°·400 on the Beckman scale. The limitations of the method are shown, and the accuracy required for concordant readings by the fact that for molecular weights above 1000, 0°·001 corresponds to nearly a difference of the increment, CH_2, but below

500 to a difference of only 2 to 4 units. Occasionally stearic acid gives abnormally high readings, doubtless caused by irregularity in the initial separation of crystals, which resisted all attempts toward correction by variation in stirring or other manipulation; but several, usually not more than three repetitions, readily revealed by concordant values, could be relied upon for the desired results. (No PP.)

In the extremely high values, 1600 or more, that define the hydrocarbons with the largest molecular weights, the observations were as closely concordant as with the oils of 300 M.W. Commercial stearic acid dried at 100° is sufficiently pure; different lots showed small variations in the constant; for example: (1) 4·431; (2) 4·467; Bernstein gives for this constant, 4·5. Particular attention was necessary in getting complete solution of the heavy oils, and these required large weights for sufficient depression.

To yield the small differences in percentages of carbon and hydrogen necessary to distinguish between the different series, the gases from the asphaltic oils require the highest temperature for complete combustion that the most infusible glass will stand with a stream of oxygen on the CuO in front of the oil. Much time was saved by weighing the bulbs filled with oxygen. Although a 50 per cent. solution of KOH was used, with solid KOH or soda lime and P_2O_5 in the safety tube of the Geissler bulb, a horizontal tube in front with soda lime and P_2O_5 invariably showed from 0·0005 to 0·0020 gram increase in weight, sufficient if lost to spoil the analysis.

Varieties of Petroleum Investigated.

General application of the method herein described to the petroleum fields of the world should doubtless involve the study of more than 100 representative varieties. In this paper is included the separation of the constituent hydrocarbons, from the following five typical crude oils:—

Table I.

Locality.	Strata.	Depth well, feet.	Sp. gr.	Per-centage at 300°, 30 mms.	Sp. gr. at 300°, vac. Residue
Cabin Creek, W. Va. ..	Lowest	1700	0·8100	25	0·8683
1st sample	Berea grit				
2nd sample	„		0·7850	20	0·8638
Rosenbury, Emblen-ton, Pa. ..	Rosenbury sand	1240	0·8080	35	0·8852
Mecca, O. ..	Loose sand	150	0·9023	80	0·9076
Sour Lake, Tex. ..	„	2000	0·9333	40	0·9580
Baku, Russia ..	„	Shallow	0·8650	35	0·9270

The Cabin Creek and Rosenbury oils are regarded as the best varieties of Appalachian petroleum, and known in the trade as

paraffin-base oils. They contain large proportions of the gasoline, kerosene and solid paraffin hydrocarbons, leaving residues solid with paraffin at 300°, 30 mm. Authentic specimens of these oils, pale yellow in colour, were procured for this examination from Mr. O. C. Dunn, of Marietta, O.

The Sour Lake oil, procured from a reliable source, is a typical heavy Southern crude, containing no hydrocarbons C_nH_{2n+2}; the crystalline hydrocarbons occasionally observed in some distillates are probably of a heavier series. (No PP.)

That the less volatile portions of the Texas oils are composed, to a large extent, of the best lubricant hydrocarbons cannot be doubted, and while the balance of the Northern crudes are of the lighter series, a large proportion in the basic Southern crudes are of the so-called asphaltic hydrocarbons which impart high viscosity to the lubricants containing them. How far the higher specific gravity and viscosity indicates superior lubricant quality depends, of course, on the inherent wearing quality of the asphaltic hydrocarbons, and this has never been precisely defined. (No PP.) In the early development of Southern oil territory, Texas oil was the synonym for high sulphur petroleum. Intimately associated with beds of sulphur, the sulphur was dissolved to the limit of saturation, and the resulting chemical changes eliminated hydrogen as hydrogen sulphide with the formation of the heavy hydrocarbons. In the formation of such heavy crudes as the Sour Lake, evidently sulphur has been a determining element. With continued production the original proportions of sulphur in these oils, one to three per cent., has been greatly reduced.

The Russian oil is a part of two barrels brought for my use 25 years ago from Baku. It is less stable than American oils, and care is necessary to avoid decomposition, even under reduced pressure. Like all Russian crudes, the distillable portion is composed of the naphthene hydrocarbons that make superior luminants, and the remainder has a smaller proportion of lubricants than American petroleum, but considerable asphaltic constituents. The great body of the Mid-Continental fields yields oils with fixed constituents; they are usually referred to as oils with a mixed base, paraffin and asphaltic, and the lubricants made from them possess a peculiar composition and quality quite different from those of the Appalachian or the Southern crudes. From the general composition of these varieties of petroleum they seemed especially well adapted for this investigation, as representing the principal fields. Mecca petroleum, specific gravity 0·9023, known as a natural lubricant since the beginning of the petroleum industry, is typical of occasionally occurring small pockets or sections at

shallow depths, where by natural agencies the original oil has been partially refined, leaving only hydrocarbons with large molecular weights, containing no gasoline, kerosene, nor paraffin hydrocarbons, and only a very small amount of the asphaltic hydrocarbons. All but 12 per cent. of the lighter end forms the best lubricants.

On account of its peculiar composition, and since there is an opportunity, for the first time, to give a description of the undecomposed hydrocarbons in a crude oil, from beginning to end, it seemed of interest to ascertain its complete composition. The lower constituents were, therefore, separated by several distillations *in vacuo*, refined, and the values obtained for specific gravity, molecular weight, and percentage composition are given in Table No. 2. The peculiar odour of some of the distillates indicate that the crude oil is not so far removed from its original organic source as the Appalachian oils.

TABLE II.

Fraction. °C.	Determinations. Sp. gr.	M.W.	C. Per cent.	H. Per cent.	Required. Formula.	M.W.	C. Per cent.	H. Per cent.	Series.	Refractive Index.
120–121	0·8600	177	86·61	13·26	$C_{13}H_{24}$	180	86·66	13·34	C_nH_{2n-2}	1·4605
130–131	0·8631	190	86·67	13·23	$C_{14}H_{26}$	194	86·60	13·40	,,	1·4625
138–141	0·8648	204	86·62	13·33	$C_{15}H_{28}$	208	86·54	13·46	,,	1·4650
150–152	0·8692	218	87·10	12·75	$C_{16}H_{30}$	220	87·28	12·72	C_nH_{2n-4}	1·4665
168–170	0·8696	226	86·87	12·85	$C_{17}H_{30}$	234	87·18	12·82	,,	1·4715
182–184	0·8722	244	87·08	12·82	$C_{18}H_{32}$	248	87·10	12·90	,,	1·4710
194–196	0·8730	258	86·95	13·12	$C_{19}H_{34}$	262	87·03	12·97	,,	1·4725
213–214	0·8750	270	86·90	12·95	$C_{20}H_{36}$	276	86·96	13·04	,,	1·4750
237–238	0·8834	334	86·65	13·24	$C_{24}H_{46}$	332	86·75	13·25	,,	1·4785
244–246	0·8840	348	86·73	13·21	$C_{25}H_{48}$	346	86·70	13·30	,,	1·4815
	Light hydrocarbons from Mecca vacuum residue.									
	0·8837	343	86·62	13·25	$C_{25}H_{48}$	346	86·70	13·30	,,	
	0·8847	389	86·70	13·25	$C_{28}H_{52}$	388	86·60	13·40	,,	

These determinations of refractive index increase with increase in specific gravity, and in molecular weight the opposite of the hydrocarbons in the Appalachian oils, and, as will appear later, even in the Mecca hydrocarbons of higher molecular weight in the vacuum residue. The distillate 244°–246° C., sp. gr. 0·8840, treated as in the separation of the D– and H– series, gave a D–hydrocarbon, sp. gr. 0·8850, refractive index, 1·4865, and an H–hydrocarbon, sp. gr. 0·8835, a lower refractive index, 1·4765, indicating more than one series, as in the higher hydrocarbons. Some of the Mecca vacuum residue that can be distilled at 300°–320° C. without decomposition, and that has been refined for use as a lubricant on fine watch and clock bearings, was separated by the solvent into the following fractions :—

	Sp. gr.	Refractive index.	The M.W. and analysis gave the following formulæ for the D-group, indicating the series, C_nH_{2n-4}:—
1–D ..	0·8837 ..	1·4835	
1–H ..	0·8780 ..	1·4805	
2–D ..	0·8789 ..	1·4815	
2–H ..	0·8710 ..	1·4765	$C_{31}H_{58}$
3–D ..	0·8705 ..	1·4815	$C_{32}H_{60}$
3–H ..	0·8682 ..	1·4755	$C_{34}H_{64}$
4–D ..	0·8745 ..	1·4785	$C_{35}H_{66}$
4–H ..	0·8680 ..	1·4760	$C_{38}H_{72}$
5–D ..	0·8750		

These hydrocarbons form the connecting link in the series between those that can be and those that cannot be distilled. The data of this examination indicates more than one series.

SERIES AND HOMOLOGOUS HYDROCARBONS IN PETROLEUM.

Investigations carried on in this laboratory and elsewhere have shown that petroleum is chiefly composed in variable proportions of the series C_nH_{2n+2}, gasoline, kerosene and paraffin hydrocarbons, of the series C_nH_{2n-2}, the light lubricants, especially of Appalachian petroleum, the series C_nH_{2n-4}, and C_nH_{2n-6}, the heavier lubricants, the aromatic derivatives of benzene, and heavier series still poorer in hydrogen to C_nH_{2n-20}, that appear in this paper, are elsewhere reported as present in European petroleum. The homologues of the heavier series above 300° C. *vacuo* appear to increase in regular increments similar to the distillable series, the D-hydrocarbons, lubricants to the final heavy ends, except in the asphaltic crudes, and the H-asphaltic of the heavy ends, in all except the Appalachian.

In the upper ends of the series first separated of all the oils examined, the specific gravity of the fractions increased very materially, some even higher than those of the lower ends. This was found to be caused by carboxylic acids or ethers more soluble than the hydrocarbons themselves. By further treatment of the upper fractions, the soluble oils were removed, leaving the hydrocarbons in Table No. 3.

The first 10–15 D– and H–homologues first separated in each crude oil were all given two or more extractions, and collected in the smaller groups presented in this table. Much time was lost in this work before it was learned that the crude oils contained more than one series of lubricants, and that the series as well as the individual homologues differed materially in solubility. While the formulæ and series represent the definite composition of the fractions separated, it should require the manipulation of much larger quantities of the crude oils than is possible in the ordinary chemical laboratory, and as in fractional distillation, a greatly prolonged treatment to isolate with a closer approximation the individual hydrocarbons. To avoid serious loss in watch glass transference, the fractions were kept in bottles saturated with the solvent, and small lots dried at 120° C. for examination.

TABLE III.

Source.	Fraction.	Sp. gr.	Determined. M.W.	C. Per cent.	H. Per cent.	Formula.	Calculated. M.W.	C. Per cent.	H. Per cent.	Series.	Refractive Index.
Cabin Creek	D-Series										
	No. 1	0·8755	309	86·76	13·21	$C_{22}H_{40}$	304	86·84	13·16	C_nH_{2n-4}	1·4920
	No. 2	0·8764	327	86·76	13·18	$C_{24}H_{44}$	332	86·75	13·25	,,	—
	No. 3	0·8815	428	86·56	13·34	$C_{30}H_{54}$	430	86·50	13·50	,,	—
	No. 4	0·8822	452	86·45	13·44	$C_{32}H_{56}$	458	86·46	13·54	,,	—
	No. 5	0·8829	488	86·32	13·48	$C_{34}H_{60}$	486	86·42	13·58	,,	—
	No. 6	0·8832	585	86·35	13·59	$C_{42}H_{80}$	584	86·30	13·70	,,	—
	No. 7	0·8835	717	86·10	13·79	$C_{52}H_{100}$	724	86·20	13·80	,,	1·4880
	No. 8	0·8855	803	85·27	13·73	$C_{58}H_{112}$	808	86·14	13·86	,,	1·4810
	H-Series										
	No. 1	0·8721	459	87·21	12·70	$C_{33}H_{58}$	454	87·22	12·78	C_nH_{2n-6}	1·4880
	No. 2	0·8725	476	87·20	12·64	$C_{34}H_{60}$	468	87·20	12·80	,,	—
	No. 3	0·8729	490	87·02	12·86	$C_{35}H_{64}$	496	87·10	12·90	,,	—
	No. 4	0·8819	635	86·52	13·51	$C_{45}H_{84}$	636	86·79	13·21	,,	—
	No. 5	0·8863	750	86·72	13·22	$C_{54}H_{100}$	748	86·64	13·36	,,	1·4870
	No. 6	0·8873	769	86·72	13·42	$C_{55}H_{102}$	762	86·60	13·40	,,	—
	No. 7	0·9063	1696	86·50	13·43	$C_{122}H_{238}$	1700	86·32	13·68	C_nH_{2n-12}	1·4810
Rosenbury	D-Series										
	No. 1	0·8796	384	86·13	13·40	$C_{28}H_{52}$	388	86·60	13·40	C_nH_{2n-8}	1·4930
	No. 2	0·8816	438	86·68	13·24	$C_{31}H_{60}$	444	86·48	13·52	,,	1·4890
	No. 3	0·8822	481	86·42	13·39	$C_{34}H_{66}$	486	86·42	13·58	,,	—
	No. 4	0·8836	518	86·15	13·71	$C_{36}H_{72}$	514	86·38	13·62	,,	1·4880
	H-Series										
	No. 1	0·8742	549	87·08	12·85	$C_{39}H_{72}$	552	86·96	13·04	,,	1·4920
	No. 2	0·8765	615	86·78	13·15	$C_{43}H_{82}$	622	86·82	13·18	,,	—
	No. 3	0·8812	639	86·65	13·25	$C_{45}H_{84}$	636	86·78	13·22	,,	—
	No. 4	0·8850	666	86·60	13·30	$C_{46}H_{86}$	664	86·74	13·26	,,	—
	No. 5	0·8848	727	86·59	13·29	$C_{50}H_{94}$	720	86·68	13·32	,,	—
	No. 6	0·8865	805	86·63	13·37	$C_{54}H_{102}$	804	86·56	13·44	,,	1·4870
	No. 7	0·8950	830	86·49	13·52	$C_{58}H_{112}$	832	86·54	13·46	,,	—
	No. 8	0·8998	980	86·69	13·28	$C_{71}H_{136}$	954	86·80	13·20	C_nH_{2n-12}	—
	No. 9	0·9079	1730	86·58	13·35	$C_{123}H_{234}$	1734	86·50	13·50	C_nH_{2n-16}	1·4870
Mecca	D-Series										
	No. 1	0·8945	465	87·37	12·61	$C_{33}H_{60}$	468	87·20	12·80	C_nH_{2n-6}	—
	No. 2	0·8950	500	87·13	12·70	$C_{35}H_{64}$	496	87·10	12·90	,,	—
	No. 3	0·8960	631	86·87	13·10	$C_{45}H_{84}$	636	86·78	13·22	,,	—
	No. 4	0·8962	662	86·60	13·20	$C_{46}H_{86}$	664	86·75	13·25	,,	—
	No. 5	0·8966	728	86·62	13·22	$C_{50}H_{94}$	734	86·66	13·34	,,	—
	No. 6	0·8982	770	86·41	13·50	$C_{53}H_{104}$	776	86·60	13·40	,,	—
	No. 7	0·8998	832	86·45	13·47	$C_{58}H_{112}$	832	86·54	13·46	,,	—
	No. 8	0·9171	1080	86·58	13·42	$C_{74}H_{144}$	1084	86·34	13·66	,,	—
	H-Series										
	No. 1	0·9058	477	87·65	12·37	$C_{35}H_{62}$	478	87·87	12·13	C_nH_{2n-22}	—
	No. 2	0·9072	550	87·57	12·48	$C_{40}H_{72}$	548	87·59	12·41	,,	—
	No. 3	0·9018	684	87·06	12·82	$C_{48}H_{82}$	688	87·21	12·79	,,	—
	No. 4	0·9022	725	87·16	12·76	$C_{50}H_{92}$	688	87·16	12·76	,,	—
	No. 5	0·9052	823	87·12	12·73	$C_{59}H_{108}$	828	86·96	13·04	,,	—
	No. 6	0·9065	992	87·22	12·75	$C_{72}H_{132}$	992	87·10	12·90	C_nH_{2n-16}	—
	No. 7	0·9600	1662	87·34	12·56	$C_{120}H_{204}$	1668	87·23	12·77	C_nH_{2n-20}	—
Sour Lake, Texas.	D-Series										
	No. 1	0·9408	450	87·93	11·91	$C_{32}H_{54}$	450	88·00	12·08	C_nH_{2n-12}	1·4980
	No. 2	0·9467	462	88·09	11·81	$C_{33}H_{56}$	464	87·93	12·07	,,	—
	No. 3	0·9482	503	87·82	12·15	$C_{37}H_{62}$	506	87·74	12·26	,,	—
	No. 4	0·9535	531	87·58	12·30	$C_{39}H_{66}$	534	87·64	12·36	,,	1·4960
	No. 5	0·9595	554	87·62	13·32	$C_{41}H_{70}$	562	87·54	12·46	,,	—
	No. 6	0·9643	849	86·80	13·18	$C_{63}H_{112}$	856	86·92	13·08	,,	1·4940
	H-Series										
	No. 1	0·9470	602	87·90	12·05	$C_{44}H_{72}$	600	88·12	12·00	C_nH_{2n-16}	1·4970
	No. 2	0·9497	630	87·60	12·45	$C_{46}H_{78}$	632	87·62	12·38	,,	—
	No. 3	0·9559	680	87·55	12·35	$C_{50}H_{84}$	684	87·72	12·28	,,	—
	No. 4	0·9643	710	87·58	12·25	$C_{52}H_{88}$	712	87·64	12·36	,,	—
	No. 5	0·9700	792	87·66	12·25	$C_{58}H_{98}$	785	87·88	12·12	C_nH_{2n-20}	1·4940
	No. 6	0·9714	854	87·50	12·24	$C_{62}H_{104}$	848	87·74	12·26	,,	—
	No. 7	0·9720	981	87·65	12·35	$C_{72}H_{124}$	988	87·45	12·55	,,	—
	No. 8	1·0230	1239	87·60	12·27	$C_{93}H_{160}$	1240	87·98	12·02	,,	—

Source.	Fraction.	Sp. gr.	Determined. M.W.	C. Per cent.	H. Per cent.	Formula.	Calculated. M.W.	C. Per cent.	H. Per cent.	Series.	Refractive Index.
Baku, Russia	D–Series										
	No. 1	0·9186	381	87·42	12·53	$C_{27}H_{44}$	368	88·05	11·95	C_nH_{2n-10}	1·4920
	No. 2	0·9251	402	87·75	11·93	$C_{28}H_{48}$	396	87·88	12·12	,,	—
	No. 3	0·9254	494	87·46	12·53	$C_{35}H_{60}$	494	87·47	12·53	,,	—
	No. 4	0·9262	640	86·91	13·03	$C_{45}H_{80}$	634	87·06	12·94	,,	—
	No. 5	0·9288	1022	87·29	12·68	$C_{74}H_{132}$	1026	86·55	13·45	,,	—
	H–Series										
	No. 1	0·9025	300	87·95	11·96	$C_{20}H_{28}$	300	88·00	12·00	C_nH_{2n-8}	—
	No. 2	0·9160	334	87·98	12·01	$C_{24}H_{40}$	328	87·80	12·20	,,	—
	No. 3	0·9167	378	87·36	12·53	$C_{28}H_{48}$	384	87·50	12·50	,,	—
	No. 4	0·9150	420	87·28	12·60	$C_{30}H_{54}$	426	87·32	12·68	,,	—
	No. 5	0·9162	460	87·29	12·66	$C_{33}H_{60}$	454	87·22	12·78	,,	—
	No. 6	0·9242	661	86·72	13·21	$C_{45}H_{90}$	664	86·75	13·25	,,	—
	No. 7	0·9360	847	86·63	13·30	$C_{60}H_{114}$	846	86·52	13·48	,,	—
	No. 8	0·9402	1008	98·31	12·68	$C_{80}H_{140}$	1100	87·28	12·72	C_nH_{2n-20}	1·4910

For the purpose of showing at a glance the consistency of the hydrocarbons described in the preceding table as they appear spread out on watch glasses, in the following table a brief description is given of the first and last members of each series from all the crude oils :—

TABLE IV.

CONSISTENCY OF THE D– AND H– HYDROCARBONS.

Cabin Creek.	Rosenbury.	Mecca.	Sour Lake.	Russian.
D–Series.				
1. Light amber. Fine, light lubricant.	1. Light amber. Fine, light lubricant.	1. Light amber. Fine lubricant.	1. Dark amber. Just flows, good lubricant.	1. Dark amber Slow flow good lubricant.
8. Dark amber. Flows readily, heavy lubricant.	8. Dark amber. Thick flow, heavy lubricant.	7. Dark amber. Thick flow, heavy lubricant.	7. Black, sticky tar. No lubricant.	8. Black, sticky asphalt, oil. No lubricant.
H–Series.				
1. Light amber. Like 1–D.	1. Thin flow, light lubricant.	1. Light amber. Thicker than CC. and Ros. No. 1.	1. Dark amber. Just flows, good lubricant.	1. Dark amber Thick flow, heavy lubricant.
8. Dark amber, thick, nearly solid.	8. Dark amber. Slow flow.	7. Black, sticky, tarry oil.	8. Black brittle solid asphalt.	8. Thick, black asphalt oil.

In the destructive distillation of Appalachian petroleum by the common method of refining the most valuable lubricants of the heavy ends, such as the last D– and H–fractions in the Cabin Greek, Rosenbury, Mecca, Table No. 3, the best lubricants in any petroleum are lost in coking. This is of less consequence in the asphaltic oils, for the lubricants in these crudes are for the most part carried over in the steam distillates, leaving only asphaltic residues.

On account of the less solubility of the lower members of each series and the separation of homologues in only one direction, it

was possible to remove very completely the higher homologues, and, therefore, to obtain data for the calculation of the formulæ of the lowest residual hydrocarbons as reliable as the methods of definition are capable of yielding. These last hydrocarbons were, therefore, carefully purified for the comparison of physical properties and lubricant value. Those from the heavier oils have the intensified qualities of the commercial asphalts; black in colour, they may be drawn out to a considerable length in very fine threads, and possess great adhesiveness. The residual lubricants from the Appalachian crudes, amber in colour, greasy in feel and of high viscosity, differ in appearance from the gray basic stocks of the Mid-Continental lubricants, which are doubtless mixtures to some extent with asphaltic bases.

On account of the limits of accuracy in the determinations of molecular weights mentioned above, the fractions with higher values, such as the Rosenbury fraction $C_{123}H_{234}$, may be incorrect by one or more increments CH_2, but by the determinations upon which it is based it must have a high value, for the fraction H–, No. 9, sp. gr. 0·8933, gave in two molecular weight determinations: (1) 1722; (2) 1718; further fractioned with sp. gr. 0·8943, it gave 1728, and still further fractioned with sp. gr. 0·9079, it gave 1730. There appears, therefore, to be no doubt as to its high molecular composition. So also the molecular weight, 1696, of the Cabin Creek H–No. 7 fraction, sp. gr. 0·9063, with the next largest value, appears to be correct, since it was separated from both specimens of the crude oil which gave fractions with the molecular weights (1) 1685, (2) 1690, and with analysis corresponding to the formula $C_{123}H_{222}$. (No PP.)

Therefore, with methane as the first gaseous hydrocarbon, and pentane as the first liquid, under ordinary pressure, passing through the several series of light and heavy liquids, through viscous lubricants and solid paraffin, the final lower end is reached in these oils, so viscous they will not flow at common temperatures, the heaviest lubricant hydrocarbons in Appalachian petroleum.

Although the Mecca H- group is composed in general of much heavier hydrocarbons than those from the Appalachian oils, the lubricants in the lower end of its series are not very different from the others. The last H–hydrocarbon No. 7, $C_{122}H_{204}$, sp. gr. 0·9600, is not a lubricant, but an asphalt. The last D-hydrocarbon, $C_{78}H_{148}$, sp. gr. 0·9171, a true lubricant, viscosity 5461 sec., and the last Rosenbury H–, No. 9, sp. gr. 0·9079, viscosity 5248 sec., water standard, 2·4 sec., at 50° C., probably the highest viscosity of any petroleum hydrocarbons, not only indicates that lower specific gravity is characteristic of the best lubricants, but it defines the difference in lubricant quality between the hydrocarbons of the

Appalachian and those of the heavy asphaltic crude oils, with the higher specific gravity of the latter. Then, further, this Mecca asphaltic oil, H– No. 7, has nearly the same high specific gravity, 0·9600, as the Sour Lake D– asphaltic oil, sp. gr. 0·9643, which resembles all the others with which it is associated, except the higher homologues, which are lubricants. The last Sour Lake oil, H– No. 8, sp. gr. 1·0230, $C_{90}H_{166}$, is a brittle asphalt for which no lubricant quality can be claimed.

The predominating asphaltic nature of the Baku oil is equally well defined, although with smaller values in specific gravity than the Sour Lake. The last H–hydrocarbon is a black, sticky asphalt, a little higher in viscosity than the Sour Lake, but the last D–No. 5, $C_{74}H_{128}$, also a black, sticky oil, has a higher viscosity than any other in this or the Sour Lake groups. (No PP.)

In the Baku oil, unlike the others, the D– series, C_nH_{2n-10}, seems to be poorer in hydrogen and heavier than the H– series. The latter appears to be composed of a large number of low molecular weight hydrocarbons, the upper lubricants, the lower asphalts. As in the Sour Lake oil, the asphaltic hydrocarbons, in part lubricants, appear to predominate; even the last D– oils are asphaltic.

Action of Reagents on the D–, and H–, Hydrocarbons Described in this Paper.

The halogens react with these hydrocarbons as readily as with those of lower molecular weights, and with the same brisk evolution of the haloid acid. Nitric acid attacks them almost with explosive rapidity, but if the first violent reaction be controlled by cooling, and heat applied, the change proceeds quietly. At about 70° C. the action proceeds most satisfactorily, with complete solution of the oil in four or five hours. At higher temperatures complete solution may take place in one hour. There is a marked difference in the appearance of the products from the D– and H– hydrocarbons. On pouring into a large volume of water, all the nitro-derivatives from the D– hydrocarbons separate in a flocculent, finally crystalline form, those from the heavy H– hydrocarbons as sticky oils. The reactions of these bodies show them to be nitro-carboxylic acids. With the ammonium salt formed by solution in ammonium hydroxide, silver nitrate precipitates the silver salt readily soluble in nitric acid. With tin and hydrochloric acid the nitro-compound is reduced to the amino-acid. Barium and lead salts are readily formed. Analysis showed a much lower molecular weight than that of the original oil. While the action of solvents indicated complex mixtures, it seems possible by proper fractionation to separate individual constituents. A study of these derivatives will be continued.

A summation of the facts relating to the nature of these hydrocarbons that make up from 25 to 35 per cent. of petroleum seems to present a well-defined distinction between the lubricant and the asphaltic hydrocarbons, and appears to support the view that the higher specific gravity of the Sour Lake and Russian lubricant hydrocarbons is due to their inherent structure, which is altogether different from the lubricant structure of the Appalachian oils. In further study now in progress of petroleum lubricants in general, including the Mid-Continental oils, the relation of high specific gravity and viscosity to wearing quality will receive attention.

Sulphur in the Sour Lake, Russian and Appalachian Hydrocarbons.

All determinations of sulphur were made by combustion in oxygen, the most accurate and expeditious method for sulphur in oils, tars and asphalts. The variation in the percentage of sulphur indicates that the solvent differentiates in the sulphur derivatives as in that of the hydrocarbons, the greater part appearing in the H– series.

Table V.

	D–Fractions.			H–Fractions.			Crude Oil,
	No. 1.	3.	6.	No. 1.	4.	8.	per cent.
Sour Lake ..	0·33	0·27	0·57	0·67	0·48	0·66	0·59
Cabin Creek ..		—			—		0·05
Rosenbury ..		—			—		0·01
Mecca		—			—		0·08
Russian		—			—		0·15

Carboxyl Derivatives in American Petroleum.

All previous records of individual fractions from the different varieties of heavy petroleum, Ohio, California, Texas, Russia, etc., have shown a wide variation and abnormally high values in specific gravity. So the different series from the heavy crudes described in this paper show similar variations. These observations indicate that the carboxyl acids, or more probably esters, are present in all varieties of American petroleum, but in very variable amounts, from the traces detected in the Appalachian oils to the two per cent. indicated by combustions of the fractions in the Sour Lake asphaltic oil. In further testing for the presence of carboxylic acids the upper D– Sour Lake fractions dissolved in ether and extracted with potassium hydroxide, the aqueous solution acidified, and again extracted with ether, leaves on evaporation a considerable amount of the oily acid residue. The specific gravity of the hydrocarbon oil before and after the extraction of No. 4–D fraction was, respectively, 0·9642 and 0·9575. The action of the solvent in carrying

up the carboxyl derivatives in the fractions of the Russian oil is plainly evident in the high sp. gr., 1·1050, of the oil extracted from the upper D–fraction, and the composition of this fraction, –C, 86·83 per cent; H, 10·41 per cent., as compared with a fraction in the middle of the series, C, 87·58 per cent; H, 12·45 per cent.; viscosity of the first oil at 50°, 317 sec., and of the second, 2005 sec., water, 2·28 sec.

The carboxyl oil dissolved out from Mecca No. 1 D–fraction with sp. gr. 1·0105 gave by combustion 86·70 per cent. C; and 12·41 per cent. H; with a difference of 0·89 per cent. for O_2. The viscosity of this oil was 468 sec., as compared with the hydrocarbon $C_{120}H_{220}$, sp. gr. 0·9171, at the lower end of the same series, viscosity 1073 sec. at 50°, water equiv., 2·8 sec. It would be of interest to isolate larger quantities of these oils and ascertain their composition.

Unsaturation as Shown by Iodine Numbers.

Of the two forms of unsaturation, open chain and the ring, evidently only the latter applies to the lubricant hydrocarbons, and it has received much attention with respect to this condition, as shown by the iodine numbers. Iodine reacts indiscriminately on the D– and H–hydrocarbons, without showing any consistent relation or differences, but with results much like those observed in distillates. Trial of the Johansen method that appears to reveal what has been regarded as addition is really substitution, not only disproved addition, but gave negative numbers to the extent of two to four units.

The Formalite Reaction.

The D–hydrocarbons described in this paper do not enter into this reaction as applied by the Marcusson method frequently quoted in works on lubrication, and the H–hydrocarbons of the Texas and Russian oils give variable mixtures with an indefinite composition. The Rosenbury fraction, 3H–, sp. gr. 0·8812, gave after the reaction 0·8827, and the Russian fraction, 4D–, sp. gr. 0·9262, after the reaction 0·9291. In no case could the reaction proceed unless the resulting increase in temperature was unchecked. No naphthene, C_nH_{2n}, lubricant hydrocarbons have appeared, and contrary to the statement of Marcusson, the hydrocarbons from American petroleum have shown a superiority in lubricant quality over those from the Russian oil.

I desire to express my obligation to my assistants, Messrs. R. C. Knapp and George Grossman, for their efficient aid in the work of this paper.

A Study of Viscosities of Fuel Oil Made from Panuco Crude Oil.*

By R. H. Brownlee, Ph.D., A.B., and W. H. Coburn.

Some three-and-a-half years ago we undertook to solve the problem of devising a method for producing fuel oil from Panuco Crude.

The prevalent suggestion at that time by refiners was to top the small amount of naphtha in the crude and run to coke enough of the crude to produce sufficient gas oil to blend with topped crude so as to furnish a fuel oil of a viscosity and pour test satisfactory for general use as a heavy fuel. This necessitated an expensive plant, with high overhead and upkeep and operating expense.

At that time, the Bunker "C" specification for heavy fuel had not been published, and the 14-16° Bé. gravity Mexican fuel from light Mexican crude was the generally accepted standard for heavy fuel.

Our problem then was to devise, if possible, a method for making a fuel oil having the required characteristics of 14-16° Bé. gravity fuel, differing only as to gravity. It seemed probable, in spite of various statements to the contrary, that the viscosity of the heavy asphaltic crude could be broken down by heating, and if this could be done without at the same time causing a separation of coke, it seemed likely that this might be accomplished without anywhere near the expense of the usual method proposed.

A few experiments were carried out, using one to three litres of Panuco crude oil, and then an equipment was made to treat ten litres at once. At first, attempts were made to heat the crude in a small still with a reflux condenser so regulated as to allow only gasoline and naphtha to distil off. This was tried, both at atmospheric pressure and at higher pressures, but was found to be unsatisfactory in that, while the viscosity was in some cases materially lowered, the temperature was not high enough under atmospheric pressure to sufficiently crack the heavy oil, and higher pressures involved too many difficulties and high expense. Accordingly, a distillation was started in a still charged with 10 litres of Panuco crude and gas heat applied so as to distil off the lighter fractions in the usual manner and under atmospheric pressure. From time to time, the gas was turned off, and after practical cooling, a measured sample was taken from the still. Heat was again applied

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until another fraction of distillate was taken from the still, and after allowing the still to cool again somewhat, another measured sample was drawn off. This operation was repeated until 60 per cent. of distillate had been taken from the still.

TABLE I.

DISTILLATION OF PANUCO CRUDE OIL.

10 Litres used. Gravity 12·4° Bé.

Temp. in Oil.	Per-centage off.	Per-centage of Naphtha to 440° F.	Gravity of Naph-tha.	Per-centage of Gas Oil.	Gravity of Gas Oil.	Viscosities at 210° F. Saybolt Universal Residue		Flash-point Cleve-land Open Cup.
						Resi-due.	and Gas Oil.	
° F.			° Bé.		° Bé.			° F.
Panuco Crude	—	—	—	—	—	—	535	135
560	5·1	4·98	53·0	0·02	—	1080	—	—
636	10·1	8·12	41·0	1·57	34·0	1885	1862	—
696	16·6	8·51	40·0	7·67	31·0	2865	1053	—
718	21·6	9·41	52·0	11·67	28·0	2100	653	—
720	26·6	10·68	51·2	15·21	27·0	2275	480	—
702	32·35	11·88	51·5	19·61	27·5	3505	512	—
710	37·83	13·01	51·5	23·81	29·0	—	540	—
710	42·41	14·21	52·0	27·11	33·0	—	830	—
710	51·01	21·29	54·6	28·61	36·5	—	3750	—
724	55·31	22·63	52·2	31·41	33·0	—	4740	—
Gravity of average sample..			51·4	—	31·5	—	—	—
Distillation loss			1·27 per cent.					

Table I. indicates the temperature in the oil during the distillation, the percentages of distillates at each stop in the operation, the percentage of naphtha up to 440° F. on basis of crude, gravity of the naphtha, percentage of heavier distillate, gravity of same, viscosity of each sample drawn from still and the viscosity of each sample drawn from still after the proportionate amount of gas oil has been added thereto. The above viscosities are plotted against the percentage of distillate off as shown in Figure 1.

The exact form of these curves will vary somewhat with the method of operation, size of stills, etc., but the general characteristics apparently hold. Similar operations with stills in which several hundred barrels were treated, indicated that the lower viscosities of the mixture of residue and distillate occur at only slightly higher percentages than here represented.

Table II. shows some of the properties of this fuel oil, while Figure 2 shows graphically the temperature-viscosity curves of a number of laboratory samples of fuel oil as compared with a regular 16° Bé. gravity fuel.

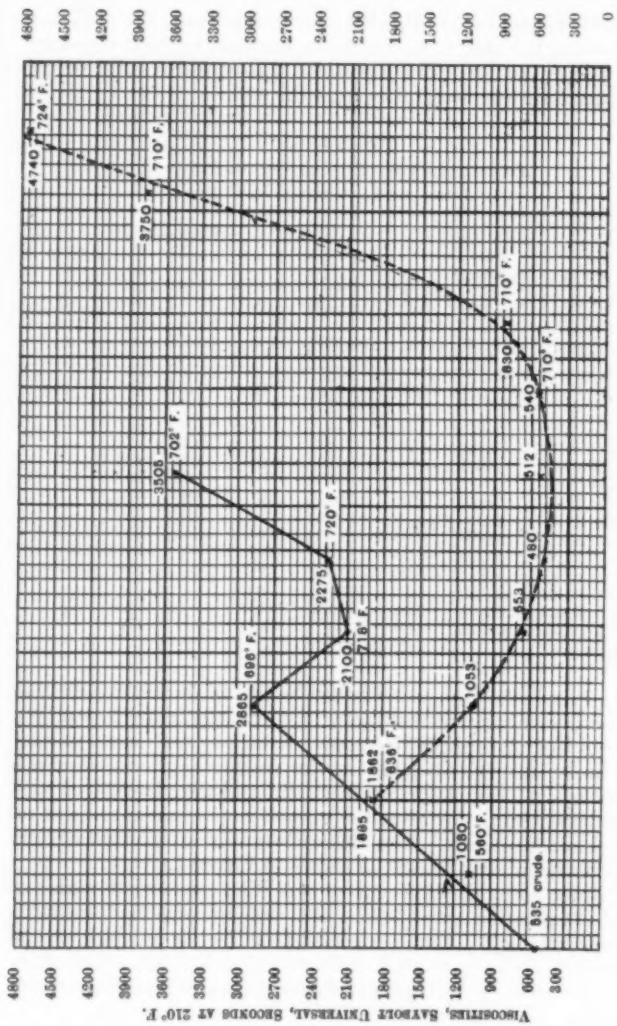

FIG. 1.

DISTILLATIONS OF PANUCO CRUDE OIL.

It will be noted that we covered a rather wide range with the laboratory samples in order to learn the possibilities. A still, 4 ft. by 12 ft., equipped with an inner stirring device, was next

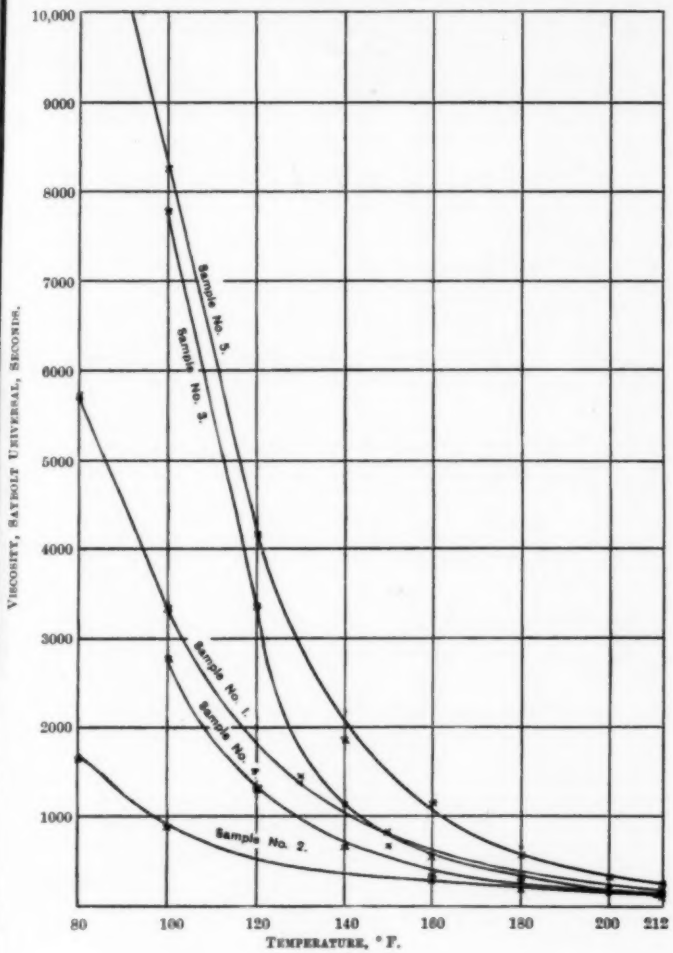

FIG. 2.

VISCOSITY CURVES.

installed and the results of a number of runs are shown in Table III. and Figure 3.

Temperature viscosity curves for not only the fuel oil made during these special runs, but also for comparison tests on two samples of

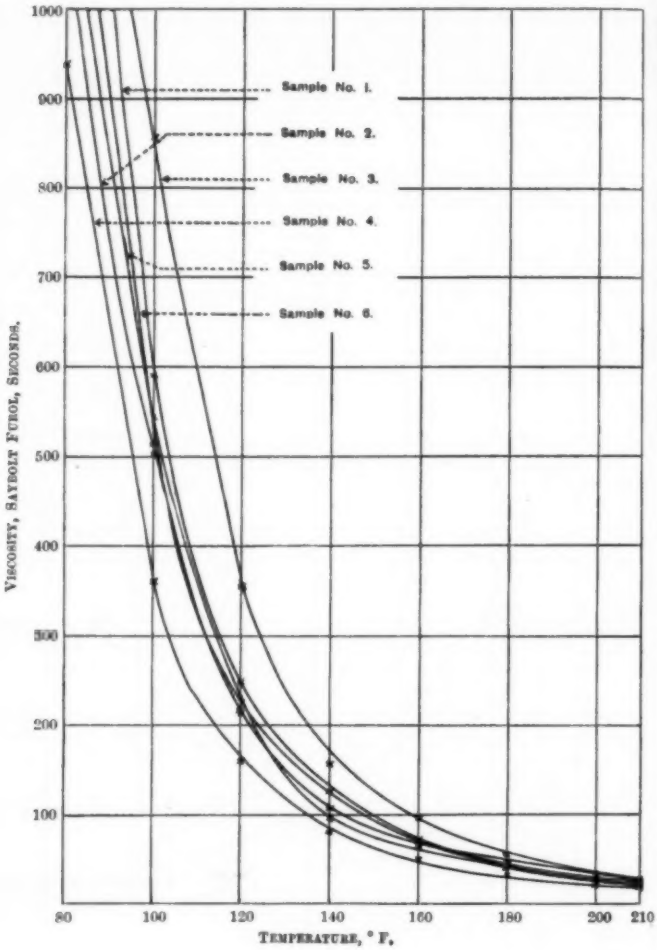

FIG. 3.
VISCOSITY CURVES.

heavy fuel oil from Light Mexican crude are shown. It will be noted by comparison of these curves that the fuel oils are analogous with respect to temperature-viscosity relationships (refer to Fig. 3).

As is generally known, the viscosity of an oil having high asphalt

content, increases quite noticeably upon standing, and it was feared that this tendency might be more marked than usual, but at the time we were convinced that no danger existed on this account.

TABLE II.

VISCOSITIES ON FUEL OIL MADE FROM PANUCO CRUDE.
Viscosities—Saybolt Universal.

Temp. °F.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Sample No. 5.
80 ..	—	1646	—	5728	37,152
100 ..	3323	897	7772	2792	8254
120 ..	—	—	3360	1319	4172
130 ..	1351	—	1445	—	—
140 ..	—	—	1121	683	1884
150 ..	808	—	666	—	—
160 ..	—	291	531	418	1129
180 ..	—	199	335	280	569
200 ..	—	159	200	185	339
210 ..	—	—	—	154	247
212 ..	180	137	124	152	238

	Residue, Gr. 11·0° Bé.	Residue No. 1 + Gas Oil, Gr. 11·1° Bé.	Residue, Gr. 11·2° Bé.	S.S. Aquitania (July, 1920), Gr. 16·1° Bé, Light Mexican.	Residue + Gas Oil, Gr. 10·6° Bé.
Naptha..	15·7%		4·5%		
Gas Oil..	4·9%				

TABLE III.

VISCOSITIES ON FUEL OILS MADE FROM PANUCO CRUDE.
Viscosities—Saybolt Furol.

Temp. °F.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4, Run No. 3.	Sample No. 5, Run No. 3.	Sample No. 6, Run No. 7.
80 ..	1358	1080	1858	938	1190	1391
100 ..	590	504	858	361	543	514
120 ..	248	213	355	160	231	219
122 ..	215	206	321	141	196	178
140 ..	125	107	157	81	98	125
160 ..	73	73	97	51	61	66
180 ..	46	47	54	31	41	41
200 ..	26	31	34	21	25	27
210 ..	24	28	28	18	22	22
Gr. ° Bé.	15·4	16·2	11·5	12·7	12·7	12·4
	Light Mexican	Light Mexican		March 24, 1921		
Gravity of crude at Newburgh .. .. 12·4° Bé.						

Commercial scale operation of the process modified to use existing equipment with minimum change was begun before the so-called semi-commercial experiments were completed. This commercial operation was entirely successful with the exception that repairs

upon still bottoms were very heavy. It is believed that a still of the general design of the one used in semi-commercial operation will largely obviate this difficulty. By constantly stirring the oil, tendency to coke is obviated. A pipe still could be used for most of the distillation, thus increasing throughput.

Plans were drawn for a 10,000-barrel refinery to be erected in England, and to be equipped with stills of this new type, but the sudden slump in fuel oil prices caused abandonment of the plans.

Several cargoes of this fuel oil were shipped abroad late in 1920 and early in 1921, with no complaint except in the case of one cargo. When this was being unloaded, it was found that the pumps

TABLE IV.

Sample No. 1, Run No. 3. Made March 23, 1921. Heated nine hours, refluxed at 720° F., 37·6 per cent. off.	Sample No. 2, Run No. 7. Made April 22, 1921. Heated four hours without reflux at 720° F., 36 per cent. off. Viscosity Furol at 122° F. = 161 secs.

	Viscosities Furol. Secs.			Viscosities Furol. Secs.	
Temp. ° F.	March 24. 1921.	Aug. 15, 1921.	May 11, 1923.	Aug. 17, 1921.	May 14, 1923.
80	938	1190	—	1391	—
100	361	543	—	514	—
120	160	231	—	219	—
122	141	196	860	178	465
140	81	98	350	125	233
160	51	61	162	66	119
180	31	41	83	41	61
200	21	25	42	27	37
210	18	22	37	22	32

would not handle some of the oil, and upon investigation there was said to have been found a thick liverlike residue on the bottoms of some of the compartments that had to be shovelled out of the ship. At the time we expected to get samples of this residue, but these were never received, and our conjecture was that probably the cold ocean water had chilled the oil and it had not been sufficiently heated by the steam coils before reaching port. We have since investigated this, however, and now conclude that doubtless there were leaks in the steam coil, or possibly between the plates of the ship, as we have found that the presence of water, whether fresh or salt, greatly accelerates the setting or coagulation of the fuel oil (see Table IX.).

A number of elements apparently affect this setting; for example, agitation, oxidation, moisture, time and temperature.

We happened to have in our laboratory a number of samples of fuel oil prepared over two years ago and hence, when we decided to publish this information some three or four months ago, we had

this at hand. Naturally, a number of ideas occurred to us that caused us to regret that several different experiments had not been started a couple of years previously, so that the results could now be tabulated. This suggested the idea of raising the temperature of the fuel being treated so as to accelerate the action and thus eliminate waiting several months for something to happen.

Table IV. shows the viscosities of two samples of Fuel Oil made early in 1921. First at the time of production, in August of 1921, and in May of the present year. Samples have stood upon the laboratory shelves during this period.

Average temperature would doubtless be 75-80° F.

We have in the laboratory, several gallons of crude from the same car as used over two years ago, and viscosities Furol of this were determined and were as follows :—

TABLE V.

VISCOSITIES PANUCO HEAVY CRUDE—GRAVITY 12·4° Bé.

Temp. ° F.	Viscosity. Furol, secs.	Temp. ° F.	Viscosity. Furol, secs.
100	1664	180	153
122	717	200	75
140	398	210	69
160	237		

This crude was treated as previously described, being heated to 720° F., taking off 36 per cent., then redistilling the distillate to strip off the naphtha, and mixing back the gas oil and pitch. This particular sample, upon distillation, showed as follows :—

TABLE VI.

	Per cent.		Per cent.
Naphtha	2·6	Pitch	61·4
Gas Oil	33·6	Loss	2·4

This gave a fuel oil having a viscosity of 160 secs. Furol at 122° F.

TABLE VII.

Temp. ° F.	Viscosity, Furol, secs.	Viscosity at 122° F. after being successively heated to the temperatures shown at left. Furol, secs.
122	160	160
140	96	170
160	55	188
180	36	201
200	25	217
210	23	245

Upon standing in laboratory for five days at an average temperature of about 80° F., the viscosity of this fuel oil had increased from 160 to 184 Furol at 122° F., and after fourteen days to 190, and upon being heated successively to the temperatures shown below and viscosity then determined at 122° F., shown as follows :—

TABLE VIII.

Temp. ° F.	Viscosity, Furol, at 122° F. secs.	Temp. ° F.	Viscosity, Furol, at 122° F. secs.
122	184	230	220
140	178	250	240
160	176	270	264
180	176	290	314
200	195	300	314
210	220		

At the same time, a sample was placed in an ice box, and after remaining there for seventeen days, still had a viscosity of 184. After fifteen days at laboratory temperature, the viscosity of this sample had increased to 186, and after 28 days at room temperature had increased in viscosity to 205 Furol at 122° F.

A fresh sample of same oil was held in oven at 180° F. and viscosities Furol were determined. The results are shown in the following table :—

TABLE IX.

Hours in oven at 180° F.	Sample No. 3. Viscosity (Furol) at 122° F. secs.	Sample No. 4. Viscosity (Furol) at 122° F. with 0·5 per cent. water added. secs.	Sample No. 5. Viscosity (Furol) at 122° F. with 0·5 per cent. of 3 per cent. salt solution added. secs.
Started	184	190	193
6	—	213	—
18	184	—	—
24	194	335	—
30	—	355	—
42	243	—	—
48	270	460	363
66	335	—	—
72	365	588	—
90	410	—	570
96	454	735	—
102	—	830	—
114	529	—	—
120	—	1009	—
138	637	—	—
162	771	—	—
168	825	1382	1002
186	1001	—	—
234	1440	—	—

Two fresh samples of this same oil were placed in flasks connected to reflux condensers heated to 180° F.

TABLE X.

	Sample No. 6. Viscosities (Furol) at 122° F. Air blown through oil.	Sample No. 7. Hydrogen blown through oil.
At start	190	207
After 6 hours	373	—
After standing 24 hours at room temperature, no blowing ..	401	—
12 hours	607	—
16 „	—	—
18 „	—	207
19 „	1065	—
42 „	—	217
66 „	—	237
90 „	—	257
96 „	4080	281
114 „	—	339
120 „	—	378
138 „	—	446

The foregoing results indicate that agitation and oxidation evidently play an important part in the above changes in viscosities of this heavy asphaltic fuel oil (see Fig. 4, illustrating tables IV., IX., X.).

In order to determine whether or not it was possible to restore fuel oil to lower viscosity after it had apparently set upon standing, the sample from tests shown in Table VII. above was taken and after viscosities from 122° up to 210° F. had been determined, the sample was again cooled to 122° F., when it showed a viscosity of 642 Furol. This sample was heated to 300° F. for one-half hour, and upon cooling to 122° F. the viscosity was 361. Heating was repeated for another half hour, when the viscosity Furol at 122° F. was 292.

Test upon this sample showed that relatively slight heating would cause a reversion to lower viscosity oil. We endeavoured to check this result, however, and while, during the first half hour, the viscosity dropped, the heating was repeated four more times, and the viscosity constantly rose instead of dropping as was expected.

Even a temperature of 400° F. failed to give consistent results, and we are finally led to the conclusion that a temperature approximately equivalent to temperatures originally used to break down the viscosity are required to get uniform results.

For comparison, some fuel oil made from heavy Argentine Crude from the Commodora Rivadavia field was heated to 180° F., and viscosities taken at 122° F. Furol. This fuel had a viscosity of 245 Furol at 122° F., when first made up, and after standing in the laboratory for sixteen days, showed 420 Furol at 122° F.

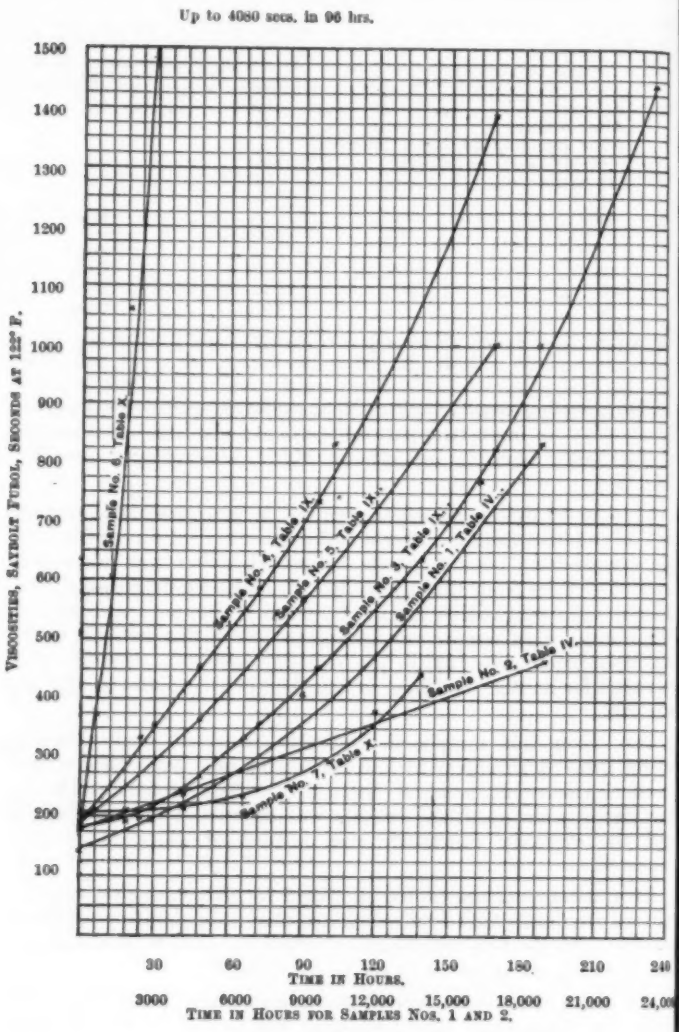

FIG. 4.

CHANGES IN VISCOSITIES OF FUEL OIL.

TABLE XI.

Temp. 180° F. Hours in oven.	Viscosity (Furol) at 122° F.	Temp. 180° F. Hours in oven.	Viscosity (Furol) at 122° F.
	secs.		secs.
—	420	96	368
6	344	102	368
24	358	126	398
30	358	174	500
48	358		

It will be noted that fuel oil from Argentine heavy crude does not as rapidly increase in viscosity as does that from Panuco, although the same tendency is shown.

In general, we may conclude from the foregoing data that high viscosity asphaltic crude oil can be converted into suitably low viscosity fuel oil by partial cracking by heat without appreciable deposition of coke. If kept at low temperature, the viscosity of this oil remains practically constant. The viscosity of this fuel increases rapidly at higher temperatures and is greatly accelerated by presence of moisture and when agitated with air.

Fuel Resources and Supplies for Road Transport.*

By Admiral Sir Edmond J. W. Slade, K.C.I.E., K.C.V.O.

Before approaching the subject of Fuel Resources and Supplies for Road Transport, it is necessary to say a few words with reference to fuel and transport generally, for it is impossible to deal adequately with the special application of fuel to road transport if we do not first consider fuel in its broad aspect.

In this country the combustion of fuel is almost the only means we possess of producing mechanical power. Water power is very little used, and under most circumstances is not applicable to anything other than fixed engines. The mechanical power obtained from the use of water power is only useful for road transport when it is converted into electricity and collected or stored by suitable means on the cars, so providing the necessary power for working the motors.

The consumption of fuel in Great Britain in 1922 amounted to 185½ million tons. Of this amount 181 million tons comprised solid fuel, coal, coke and briquettes, and 4½ million tons comprised liquid fuel, crude oil, fuel oil, gas oil, kerosene and petrol.

Of the solid fuel, manufacturers and general purposes consumed 91 million tons; domestic consumption, 35 million tons; gas, 15 million tons; electricity, 7 million tons; railways, 13 million tons; and bunkers, 20 million tons.

Of the liquid fuel, road and aerial transport consume about 1½ million tons; agricultural requirements, fishing, industrial purposes and lighting, ½ million tons; bunkers, 2 million tons; general purposes, ¼ million tons.

So far as regards road transport, therefore, we are concerned with only 1½ million tons of fuel out of a total of about 190 million tons which are consumed yearly in the United Kingdom.

Since, however, the majority of the engines which use liquid fuel are internal combustion engines, it is impossible to substitute solid for liquid fuel except in the cases where liquid fuel is burnt under boilers for the purpose of raising steam.

Coal is used to a certain extent as fuel for road transport, but statistics as to the amount so used cannot be given; the quantity,

* Paper read before, and published by permission of, the Road Traffic Conference, Commercial Motor Transport and Roads Development Exhibition, 1923.

however, is not great, and since it is all produced in this country we are not likely to suffer from any shortage in the near future.

The principal fuel used for road transport is, as we all know, one or other of the various products of petroleum, and as petroleum is not found in commercial quantities in these islands, it is a matter of considerable interest to us to know whence our supplies are derived.

Since the fuel mainly used at present for road transport is merely a product of petroleum—namely, petrol—we must, in order to get a true picture of what is taking place, consider the question from the broad standpoint of the supply of petroleum rather than from the narrower one of the supply of motor spirit, for, in order to obtain petrol, we have to produce the other main derivatives of petroleum.

A large amount of petroleum is imported into the United Kingdom, either as crude oil or in a semi-manufactured condition, in order that it may be refined here and distributed in the particular form in which it is required as fuel for road or aerial transport. Attention is drawn in the first place to Table A, which gives in millions of tons the actual production of petroleum by the various oil-producing countries.

For the purpose of comparison the production of each country in the year preceding the war is shown as well as the production in the last year of the war and in the four complete years which have elapsed since the war.

The two first columns are more or less a matter of interest, but what really concerns us is the present production of petroleum and how it is changing, and for the purpose of elucidating these points attention is directed principally to the figures of the last four years.

In order to make it clear where our petroleum supplies are derived from the chief oil-producing areas of the world have been marked on the map, Fig. 1, showing in broad outline the geographical situation of our principal sources of supply. No attempt has been made to indicate the actual fields, because to do so would introduce details which tend to obscure the main points.

You will observe from the map that the American oilfield is situated principally between the approximate latitudes of 20° and 40° N. This area lies within the United States and Mexico, and is usually treated as two separate areas in most statistical tables; but since it is actually one producing area and the interchange of petroleum products between the two countries is constant and complicated, the field is treated as one source of petroleum.

Similarly, oil produced in the East Indian Archipelago comes from various islands which are not all under the same sovereignty, but nationality is ignored and the production from all these islands

treated as coming from one main source. It will simplify the consideration of the problem under discussion if we deal with the sources of production on a geographical rather than on a national basis.

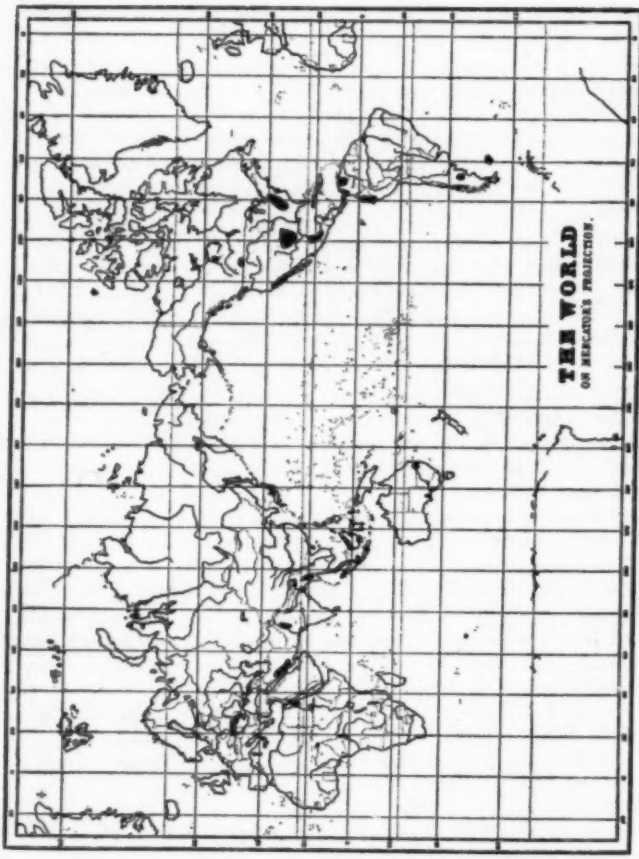

FIG. 1.
The shaded portions indicate the chief oil-producing areas of the world.

Turning again to Table A, showing the production of petroleum in the world, the figures at first sight appear to be very disturbing, for we would imply from them that the United Kingdom is absolutely dependent on the goodwill of one country only for the

supply of a commodity which is essential to the continuance of her economic life and therefore to her prosperity as a nation.

It must be remembered that without petroleum and its products road transport in its modern development and all forms of aerial transport would cease and that our marine activities, both commercial and naval, are becoming more and more dependent on uninterrupted supplies of petroleum.

TABLE A.

World Production of Crude Petroleum and Percentages of Total produced in each Area. Millions of Tons. Tons of 2240 lbs.

Area.	1913. Total.	1913. Per cent.	1918. Total.	1918. Per cent.	1919. Total.	1919. Per cent.	1920. Total.	1920. Per cent.	1921. Total.	1921. Per cent.	1922. Total.	1922. Per cent.
U.S.A. and Mexico	39·2	71·0	56·9	81·3	63·3	84·6	82·4	87·4	91·5	87·3	101·9	86·6
Rumania.. ..	1·9	3·4	1·2	1·7	0·9	1·2	1·0	1·1	1·1	1·0	1·3	1·1
Russia	9·0	16·3	5·5	7·9	3·5	4·7	3·5	3·7	3·9	3·7	4·8	4·1
E.I. Archipelago	1·5	2·7	1·8	2·6	2·2	3·0	2·6	2·7	2·5	2·4	2·7	2·3
British India ..	1·1	2·0	1·0	1·4	1·2	1·6	1·0	1·1	1·1	1·0	1·1	0·9
Persia	0·2	0·4	1·0	1·4	1·3	1·7	1·6	1·7	2·2	2·1	2·9	2·5
All other areas ..	2·3	4·2	2·6	3·7	2·4	3·2	2·2	2·3	2·6	2·5	3·0	2·5
Total ..	55·2	100	70·0	100	74·8	100	94·3	100	104·9	100	117·7	100

Authority: Official statistics, Russia, estimated in part. Total from U.S. Geo. Survey.

The importance of petroleum does not cease with its use as fuel, because it forms the basis for most lubricating oils for modern fast-moving machinery, and a failure of our supplies of petroleum would mean, therefore, complete dislocation of all our factories, railways and industries in which machinery plays so vital a part.

It follows that it is of the utmost importance that we should be able to feed ourselves independent of any one State, however friendly, for the supply of petroleum, for in a case of emergency the temptation to " twist the lion's tail " by holding up supplies of this essential commodity is not one that should be put in the way of any nation if by any means in our power we can avoid such a possibility.

The situation, although not wholly satisfactory, is not quite so precarious as it appears at first sight, for we have to remember that the factor which most affects us is not so much the total production of petroleum of any given country, as the amount of that production which is available for export—namely, the " exportable surplus."

In Table B is shown the total amount exported from each oil-producing area and the percentage of the total production of each area which is thus available for export.

From this table we see that the proportion which is available for export is comparatively small in the case, respectively, of the American, Russian and British Indian areas, while it is very large in those of the East Indian Archipelago and the Persian areas. It

follows, therefore, that any increase in the production of the latter group has much more importance from the point of view of the British consumer than a similar increase would have in the case of the production of the former group.

The internal consumption of Russia has always absorbed the majority of her production, and India takes practically the whole

TABLE B.

Amount of Petroleum (all Grades) exported from each producing Area (Millions of Tons) and Percentages exported of Total Production in each Area. Tons of 2240 lbs.

Countries.	1913. Amt.	1913. Per cent.	1918. Amt.	1918. Per cent.	1919. Amt.	1919. Per cent.	1920. Amt.	1920. Per cent.	1921. Amt.	1921. Per cent.	1922. Amt.	1922. Per cent
U.S.A. and Mexico	7·391	20·0	12·137	21·3	13·196	20·8	18·150	22·0	19·303	21·1	20·493	20·1
Rumania	1·036	56·6	—	—	0·039	4·4	0·257	25·5	0·362	32·0	0·430	31·9
Russia	0·442	5·2	—	—	—	—	—	—	0·042	1·1	0·185	3·8
E.I. Archipelago	0·900	58·0	1·218	66·0	1·905	88·0	2·070	85·0	1·466	62·3	1·308	56·3
British India ..	—	—	0·075	7·0	0·119	10·3	0·063	6·1	0·061	5·8	0·063	6·0
Persia	0·164	60·0	0·880	81·3	0·983	72·6	1·243	70·3	1·864	80·7	2·468	84·7
Totals ..	9·933		14·310		16·242		21·783		23·096		24·947	

Authority: Official Statistics.

of the production of the British Indian fields; but this has not been so in the case of the United States, and the advent of large competing production in fields where the exportable surplus is large forms a disturbing factor in the oil politics of the western hemisphere.

Towards the end of the war it was the cry in America that consumption was overtaking production and that the end of the economic life of the American oilfields was in sight. But since then new and apparently prolific oilfields have been discovered in California and Oklahoma, with the result that, notwithstanding an enormous increase in home demand, production is now in excess of consumption, stocks are increasing, and prices have fallen. How long this state of affairs may continue it is impossible to predict, but the steady increase in the quantity of oil coming forward at competitive prices for the European market from fields other than the United States and Mexico will certainly tend to force up the accumulation of stocks until the producers agree to adjust their production to requirements.

Referring again to the table (B), which shows the participation of each area in the world's oil market, we see that the share of each in 1913—the year before the war—was as follows:—

	Per cent.
American area	75
Rumania	11
Russia	4
East Indian Archipelago	9
Persia	1

Last year, 1922, the figures were as follows :—

	Per cent.
American area	82
Rumania	1·8
Russia	0·8
East Indian Archipelago	5·2
British India	0·2
Persia	10

In other words, the United States, Mexico and Persia have absorbed the share in the world's markets which has been lost by Rumania, Russia and the East Indian Archipelago.

Let us now deal with the more special case of our home market in the United Kingdom as being that in which we are directly interested, though at the same time it is important to have before us a general statement of the world position, because there is such close connection between the various markets that nothing can happen in one without the others being more or less affected.

The next table (C) shows the consumption of all grades of petroleum products in the United Kingdom for the same series of years, totals and percentages.

TABLE C.

U.K. Consumption of Petroleum Products. Quantities and Percentages of Total derived from each Area. All Grades. Tons of 2240 lbs.

Countries.	1913.	Per cent.	1918.	Per cent.	1919.	Per cent.	1920.	Per cent.	1921.	Per cent.	1922.	Per cent.
U.S.A. and Mexico ..	1,234,834	60·2	4,445,281	81·9	2,280,020	78·9	2,981,451	84·1	3,478,784	75·9	2,951,715	63·7
Rumania ..	220,618	10·8	—	—	—	—	2,357	0·1	9,816	0·2	45,388	1·0
Russia ..	138,851	6·8	—	—	2,957	0·1	21,527	0·6	6,230	0·1	25,585	0·6
E.I. Archipelago ..	137,503	6·7	108,888	2·0	115,046	4·0	121,603	3·4	131,500	2·9	204,921	4·4
British India ..	48,323	2·3	18,107	0·3	52,242	1·8	27,286	0·8	8,993	0·2	14,640	0·3
Persia ..	6,365	0·3	1,783	0·1	83,756	2·9	151,302	4·3	635,645	13·9	1,094,403	23·6
Other sources..	264,604	12·9	852,066	15·7	354,758	12·3	240,927	6·7	314,414	6·8	294,803	6·4
Total ..	2,051,098	100	5,426,125	100	2,888,779	100	3,546,453	100	4,585,382	100	4,631,455	100

Authority: Customs and Excise, except for Persia.

The points to be particularly noted are: First, the great increase, 2,580,000 tons, in imports between 1913 and 1922, a period of nine years, or an increase of 125 per cent. This increase has been provided, for the most part, by the American and Persian areas; but the share secured by Persian oil is greater than that secured by American oil, as I think you will see from Fig. 2.

The Russian and Rumanian contributions are omitted, as they are comparatively negligible.

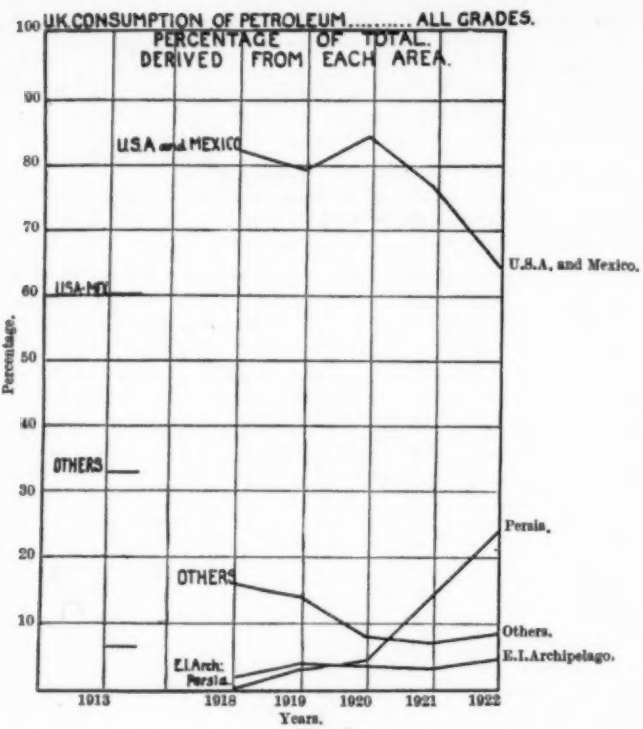

FIG. 2.

TABLE D.

U.K. Consumption of Motor Spirit. Quantities and Percentages of Total derived from each Area. Tons of 2240 lbs.

Countries.	1913.	Per cent.	1918.	Per cent.	1919.	Per cent.	1920.	Per cent.	1921.	Per cent.	1922.	Per cent.
U.S.A. & Mexico	74,944	20·9	501,434	76·3	406,673	60·3	435,982	61·5	522,153	55·1	537,172	46·4
Rumania ..	18,779	5·2	—	—	—	—	2,357	0·3	9·816	1·0	1,430	0·1
Russia	48,797	13·6	—	—	—	—	4,038	0·6	—	—	—	—
E.I. Archipelago	137,418	38·2	105,063	16·0	115,046	17·1	121,603	17·2	131,500	13·9	202,698	17·5
British India ..	48,323	13·4	18,107	2·8	52,242	7·7	27,286	3·8	8,993	0·9	14,629	1·3
Persia	—	—	698	0·1	59,329	8·8	84,273	11·9	230,128	24·3	348,258	30·1
Other sources ..	31,389	8·7	31,602	4·8	41,346	6·1	33,429	4·7	44,927	4·8	53,463	4·6
Total ..	359,650	100	656,904	100	674,636	100	708,966	100	947,517	100	1,157,650	100

Authority: Customs and Excise, except for Persia.

The next table (D) constructed similarly to those we have seen, gives the import of motor spirit, and is still more remarkable,

as it shows an increase of 798,000 tons, or a percentage increase of just over 200, the greater part of which has again been provided by the American and Persian areas. Fig. 3, however, brings out very clearly the fact that the increased participation secured by the American area took place during the war period, and since then the American share has been falling at almost the same rate at

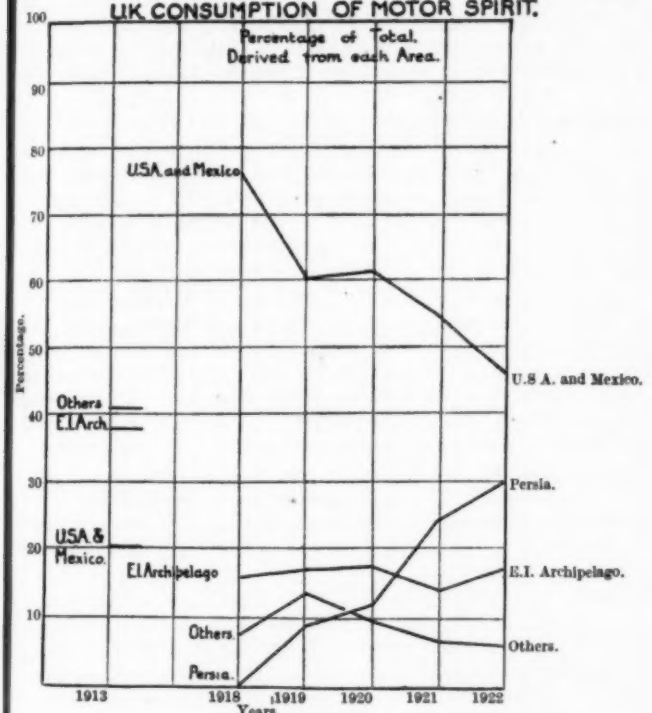

FIG. 3.

which the Persian share has been increasing, and the amount of the supplies from the two areas is rapidly approaching equality.

In 1913, before the war, when the consumption of motor spirit was much smaller than at present, the respective proportions held by the producing areas were as follows :—

	Per cent.
American area	21
Russia	14
Rumania	5
East Indian Archipelago	38

The shares of the principal producing areas last year (1922) were as follows:—

	Per cent.
American area	46
East Indian Archipelago	17
Persian area	30

The home production of motor spirit from shale, which showed quite a respectable participation of 8 per cent. of the home market in 1913, holds to-day only under 2 per cent. of the market, although the total production has only dropped from 29,000 tons to 20,000 tons.

To sum up this cursory survey of the position of motor spirit as it appears to-day, the two areas which supply the bulk of the spirit used in this country are the American and the Persian areas, and although at the present moment the greater part of the supply comes from America, it looks as if in the future—if we may be guided at all by the evidence of the graphs—we shall be drawing more of our supply from Persia than from America.

The proportion coming from the East Indian Archipelago appears to be more or less constant, while Russia and Rumania for the moment do not count for very much. Should the Russian fields be opened up again in the near future, it is doubtful whether they will have any appreciable effect on the market here. In the past Russia has absorbed the greater part of what she can produce, and it is unlikely that different conditions will obtain in the future.

Similarly, the Rumanian area is not capable of seriously participating in the market here; her production is largely taken up in her local markets, and by that is meant not only Rumania herself —who absorbs 60 per cent.—but Galicia, Austria and the countries bordering on the Danube.

The production in the East Indian Archipelago is rising, but it is not rising at the same rate as the demand for motor spirit here, and therefore it is only to be expected that the share of the market which this area held before the war should have fallen. It is, however, the most important source of supply after the American and Persian areas.

Let us now turn to the question of consumption. Within certain limits it is consumption which stimulates production, and it is therefore essential that it should be considered in connection with supply.

The table (E) gives the number of motor vehicles in the United Kingdom and in the United States for the last five years. From this it will be seen that while they have increased 62 per cent. in this country during that period, they have increased 100 per cent. in the United States during the same time.

TABLE E.

Motor Vehicles (including Passenger Cars, Trucks and Motor Cycles).

Year.	United Kingdom.	United States.	Canada.	Others.	World.
1919	620,000 (Est.)	7,596,503*b*	341,396*e*	—	—
1920	645,000*a*	8,887,572*b*	407,064*e*	—	—
1921	826,700*a*	10,505,606*b*	470,862*f*	3,702,526	15,505,748*c*
1922	921,321*a*	12,567,376*c*	516,307*f*	—	—
1923	978,000*a*	14,652, 25*d*	—	—	—

Authority: *a* Ministry of Transport figures of licences current on a certain date. *b* Automotive industries. *c* U.S. Commerce Reports. *d* *Oil and Gas Journal.* *e* Royal Statistical Society. *f* *Board of Trade Journal.*

As a rough estimate it is assumed that the annual consumption of motor spirit for each vehicle is about 352 gallons in the United States and about 330 gallons in the United Kingdom. This estimate is not absolutely reliable, but gives us a guide as to the increased demand for motor spirit which is likely to be made owing to the increase in the number of motor vehicles licensed.

A more reliable estimate can be arrived at from the results of running an ordinary commercial fleet of motor vehicles, which comprises every type, from the 5-ton lorry to ordinary cars. By taking the results of each type and working out the averages, properly weighted, in order to arrive at the most accurate result, we find that the consumption of petrol for a ton-mile is about 0·09 gallon.

It is interesting to compare this with the results obtained afloat, which give, according to data published in the *Motor Ship*, the consumption of fuel for each ton-mile as :—

Boilers coal fired	0·0349 lbs.
Boilers oil fired	0·024 lbs. =0·0027 gallons
Internal combustion engines	0·0082 lbs. =0·0009 gallons

The increase in the number of vessels using petroleum in one form or another as fuel is very great, the result of this being an increased demand for petroleum products, which in its turn stimulates production everywhere.

So far motor spirit only has been dealt with as a fuel, but kerosene and fuel oil are also used for that purpose to a less extent.

The imports of kerosene into the United Kingdom in 1913 were 635,000 tons and they are now 719,000 tons, an increase of only about 13 per cent., which is much less than might be expected, taking into consideration the increase of population during that period. Of this total :—

	Comes from
68 per cent.	America
6 per cent.	Rumania
3 per cent.	Russia
20 per cent.	Home resources (mostly manufactured from Persian oil, imported either as crude or semi-refined)
3 per cent.	Other sources

These figures are practically the same as before the war, with the exception that Rumania's share was then 14 per cent. and the home production only a fraction of 1 per cent.

Of this amount about 57,000 tons, or 8 per cent., is used for transport purposes; 517,000 tons, or 72 per cent., for illuminating and heating; and the remaining 143,000 tons, or 20 per cent., for general industrial purposes.

With regard to the use of fuel oil for transport purposes, very little has yet been done in this matter. Research is proceeding, but up to the present no really economical commercial method of utilising fuel oil on the road has been evolved.

The imports of fuel and gas oil amount to 1,800,000 tons, practically the whole of which is used for either industrial purposes, for bunkers, or for gas making.

As has already been pointed out, the importance of petroleum does not end with its use as fuel, and as the basis of nearly all lubricating oils is petroleum, the question of the sources of supply of lubricating oil cannot be separated from that of any other product of petroleum. In the figures already given for the import of petroleum lubricating oils have been included.

It is estimated that last year (1922) the world's consumption of lubricating oils was about 3½ million tons, but as many of these oils are compounded and not straight-run oils it is difficult to give any figure for the amount of petroleum products consumed thereby; but it is known that this figure is very large. Worked out on the results of the running of a large fleet of commercial vehicles, on a total distance run of about 1½ million miles, the consumption is approximately 0·006 gallon per ton-mile, in round figures.

An oilfield is a wasting asset and its life is as strictly limited as the life of a coal mine or any other mine, and as at the same time the demand for petroleum becomes greater every day, it is no matter for surprise that the search for new sources of supply is very insistent. Certain of the oilfields in the United States which were among the first to be developed have already passed their zenith as producing fields, and are showing distinct signs of depletion.

In the search for new areas, the two which hold out the greatest promise at present are the Southern and Central American area and the Iraq and Persian areas.

In the four years since the war the output of Peru has increased from 300,000 to 700,000 tons, and that of Venezuela from 50,000 to 350,000 tons, and a very considerable increase on these figures may be looked for in the future. It is known that there are areas in both these countries which are very promising, but which have not yet been developed.

It is reported that in Bolivia there are also promising signs of oil, but the country being covered by dense forests, the geological features are much hidden and it is very difficult to explore properly. It is also so far from the sea that its exploitation would be most expensive, and it is improbable that any serious attempt will be made to open it up until the more accessible fields have been thoroughly explored.

Colombia is another very promising area which has yet to be developed. The quality of the oil is good, and again it is to a great extent the physical difficulties of the country which are holding up development of these possible fields of supply.

The natural outlet for the oil from these areas is the American Continent and not Europe. The increase of demand in North America will be followed by an increasing demand in South America, and, as in all other fields which are within easy reach of densely populated industrial countries, the near-by market naturally absorbs the greater part of the production and only the surplus, if any, is available for more distant sale.

In the African Continent the prospects of oil so far do not appear to be good. There is said to be a promising field in Angola, which is being developed by American interests, and there are signs of oil in other parts; but up to the present no indications are available from which to infer the presence of a large producing field.

The Egyptian field has been disappointing to a certain extent, and it still remains to be proved whether it extends south into Abyssinia and the Soudan.

In Asia lies the principal hope of an expansion interesting to the European market.

In Persia the prospects of oil are extremely good. The proved fields are very prolific and there are encouraging signs of oil over large areas which have yet to be investigated. In fact, there is little doubt that in the near future Persia will be able to produce all the petroleum required by the United Kingdom.

In Mesopotamia—or Iraq, as it is now officially named—there are indications of a very large field, but it will be some years before this can be proved and brought into production, and a large amount of capital would have to be provided and expended for the purpose. Pipe-lines and refineries capable of dealing with large quantities of oil are not undertakings which can be installed at short notice. Distances look small on most maps of Iraq, but as a matter of fact it is a country larger than the whole of France, and to bring the oil from the fields to the Mediterranean would require a pipe-line sufficiently long to go from Madrid to Paris, and the distance

from the present only proved field at Naft-Khana to the head of the Persian Gulf is as far as from Calais to Marseilles.

The fields in the East Indian Archipelago remain fairly constant in production, but there has been a considerable increase in the last four years in the output of the field at Sarawak—namely, from 71,000 tons to 406,000 tons. There is also a promising field in Sumatra, which is now in process of examination.

In Europe the only field of any importance at present is the Rumanian. The effect of the war is still being felt and the production has not yet reached the level it attained before the war. In 1913 it amounted to 1,800,000 tons and in 1922 it was only 1,300,000 tons; but, as already pointed out, we cannot expect to draw any considerable portion of our supplies from these fields, because the output is largely absorbed by the local markets before it can come to this country.

The fact that at the present moment we are practically dependent on foreign countries for the supply of liquid fuel of all kinds renders it a most important question to find a substitute more under our own control.

Research is busy on the elucidation of this problem, but one is bound to say that, looking at the question as a whole, no satisfactory solution has yet been found. There are three lines of attack:—

1. The modification of existing known light hydrocarbons other than petrol to suit the present internal combustion engine without submitting the latter to radical changes. As examples of this we may consider benzole, alcohol and various mixtures of these with each other or with other light hydrocarbons.
2. The modification of existing engines to use liquid or other substances which are not at present employed as fuels for road and aerial transport. As an example of this we may take the utilisation of heavy hydrocarbons, such as ordinary fuel oil or of coal in a powdered form, or of both in combination.
3. The discovery of some entirely new method of producing mechanical power.

Of all these the most effective and promising substitute at the moment is benzole. It is largely employed on the road, and with suitably adjusted carburettors gives every satisfaction to its users. In fact, it is claimed that it is preferable to petrol for many purposes and gives greater efficiency. It is not proposed to discuss the advantages of benzole, either by itself or as a mixture with petrol or other hydrocarbons, because it cannot be adequately dealt with in a general review, but should be treated as a separate subject.

The fifty-fifty mixture is now a standardised commercial article and appears to give every satisfaction.

Benzole is a product of coal and is obtained from coke ovens and gas plants, and it is estimated that, on an average, 2½ gallons of benzole are obtained from each ton of coal. If we could get this average from all the coal retorted in this country it would go a very long way towards securing our position as regards motor fuel, but the production of benzole must always be limited by the demand for the other derivatives of distillation, chiefly coke.

There is also the possibility of obtaining liquid fuel from the retorting of all coal used in this country. At present, although there are numerous retorts working, it does not seem as if the industry was yet out of the experimental stage in so far as the production of commercially utilisable motor fuel is concerned.

Motor fuels, of whatever nature they may be, compete with each other on the basis of price and efficiency, and, given approximately equal efficiency, it is the price which rules. The basis price is fixed by the price of petroleum liquid fuels of similar quality, and unless the undertaking can produce and market its fuel at a price equivalent to the basis price it has no chance of establishing itself on the market. The trouble in most cases is to dispose of the by-products, and it is this difficulty which causes the price to rise. Then again, the quality of the crude oil produced in the retorts is often such as to present great difficulties in the ultimate treatment; this again sends up the price, and until the processes are so developed that the refiners are supplied with a crude oil which is more or less consistent in composition this difficulty will remain a serious one.

On the face of it the proposal that no coal should be used in its crude state is thoroughly sound, as much calorific value is now undoubtedly lost in the unscientific use of the fuel. The subject, however, does not appear as yet sufficiently explored to warrant compulsory powers being asked for, as has been suggested in various quarters.

As another possible substitute for petroleum products we have alcohol, either alone or as a mixture with other volatile fuels. Here again research is busy, but so far, although laboratory results are hopeful, success on a large commercial scale has not yet been achieved. There is one well-known brand of an alcohol-benzole-petroleum mixture on the market.

The raw materials for the manufacture of alcohol on a commercial scale exist within the Empire, and it depends upon the successful prosecution of present research whether it becomes a practical proposition or not. The chief difficulty, however, and one that has so far not been solved, is to find a sufficiency of suit-

able raw material within a given area to render commercial exploitation profitable.

Then again, it is suggested that alternative fuels may be obtained by the hydrogenation of some of the heavier hydrocarbons, such as solid naphthaline; but it is understood that, although used in Germany, such fuels are not likely to be used alone, but only as a mixture with other materials.

In discussing the merits and demerits of various fuels we are apt to miss the importance of the general question. What we require is something which is readily portable and when subjected to a simple chemical process increases its bulk more or less rapidly or causes gas or vapour to increase its bulk. By this means we can at will transform the readily portable material into a means of producing mechanical power. So far the only range of substances which have been found to be commercially suitable for this purpose are the hydrocarbons.

The hydrocarbons are used in either solid, liquid or gaseous forms and on combustion produce gas at a high temperature, occupying many times the volume of the original hydrocarbon. This phenomenon takes place with varying degrees of rapidity and is called by different names according to the speed with which the transformation takes place. The phenomenon is the same in the general scheme of its chemical changes, whether we burn coal to produce heat to raise steam or explode a petrol mixture inside a cylinder to drive a piston, or detonate a charge of gelignite to throw down a mass of rock in a quarry, or detonate a bomb dropped from an aeroplane to destroy half a city. The difference is only one of time.

We possess in this country an enormous supply of the best type of solid hydrocarbon—namely, coal—but we do not possess the only natural liquid hydrocarbon at present known, which exists in large quantities—namely, petroleum.

By the progress of engineering science we have produced an engine using liquid hydrocarbon, which is far more economical and efficient for certain purposes of transport than engines using solid hydrocarbons, and we have thereby made ourselves entirely dependent upon adequate supplies of petroleum being forthcoming.

It is absolutely essential for the progress of our national life that under no circumstances should our transport in any of its numerous phases be interrupted. If this should occur, there is no need to picture what the result would be in peace, and in war it would mean immediate annihilation.

We cannot stop the progress of science and it would be folly to hope or try to do so. We welcome the introduction of more and

more efficient methods of producing and employing mechanical power; what we have to realise, however, is that we cannot sit down with equanimity and see the means of producing this mechanical power pass out of our control into the control of others. It would be like possessing the lower of two mills on a stream, where the man who holds the upper mill is at liberty to cut off the water supply from the lower whenever he wishes.

At the present moment the position is that we are absolutely dependent on adequate supplies of petroleum. There is no prospect that these will not be forthcoming under normal circumstances, but the control is not in our hands. The wells from which the petroleum comes lie for the most part in foreign countries, and in a very large measure the control of this supply is also in foreign hands.

At the same time it is known that under certain conditions solid hydrocarbons, of which we possess an ample supply, can be broken up and will yield products which can be used in internal combustion engines, but so far this has not proved a commercial success.

In view of the extreme importance of securing under all circumstances the supply of adequate quantities of suitable liquid fuel for transport purposes a national policy is clearly indicated—namely, to consolidate such control as we possess over existing oilfields and to push forward research work to perfect the processes by which our ample store of solid hydrocarbons can be converted economically into the all-essential liquid form. If this can be accomplished, not only shall we secure an alternative source of supply of fuel for motor transport, but we shall effect very considerable economies in the use of our coal.

If we look at the figures which were given at the opening of this paper we can form some sort of an idea of the importance of these possible economies. Further, not only shall we be able to utilise in a more economical manner fuel actually now burnt, but we shall be able to bring into use other forms of coal which are at present unusable.

It must not be assumed that the development of this industry will destroy or even seriously injure the petroleum industry. The advent of electricity was said to be going to destroy the gas industry, but both now flourish side by side. Wireless telegraphy was said to be going to ruin all those who hold shares in the great cable companies; it is found, however, that there is ample room in the world for both, and wireless has done more, for it has provided a means whereby we can, in case of necessity, secure the continuity of our communications free from all danger of interruption.

In the same way the same results will be attained in the case of fuel. If we can develop the production of liquid fuel on com-

mercial lines from solid hydrocarbons we shall render the fuel position for our essential services safe in case of necessity and we shall not do any serious damage to the petroleum industry. There is ample room in the world for both industries, and it is for us as a nation vitally dependent on transport to bear in mind the immense importance of this question in its broad aspects, and by using every effort to extend our influence in the one industry and to develop the other to consolidate thereby our fuel resources and supplies for road transport.

DISCUSSION ON MR. E. H. CUNNINGHAM-CRAIG'S PAPER:

"Some Recent Researches Bearing Upon the Origin of Petroleum."

Mr. T. Sutton Bowman wrote :—Arising out of Mr. Cunningham-Craig's paper, it will be recalled (see paper by Dr. Hume*) that in the Hurghada area, from which practically the whole of Egypt's oil production is now coming, oil is found in three main zones :—

(*a*) From horizons in the Gypsum series.

(*b*) Associated with the unconformity at the base of the Miocene.

(*c*) In Cretaceous sands below the unconformity.

The oil from (*c*) is a heavy asphaltic oil, with sp. gr. over 0·9, whilst the oils from (*a*) and (*b*) are considerably lighter. The bulk of the production is from (*c*).

The Cretaceous production is from sands interbedded with carbonaceous and micaceous shales. Carbonaceous shales of Cretaceous age are of widespread occurrence in Egypt, though no workable coal is known to exist despite exhaustive search.

In borings at Gebel Tanka, in Sinai, oil in small quantities was found just above lignite seams, which in turn had gas below them. The age of these beds was definitely determined by fossil evidence as Cenomanian (see "Geography and Geology of West Central Sinai," by Dr. John Ball, pp. 204 *et seq*).

Further down in the same bore, in Nubian Sandstone, thin coaly seams overlay traces of oil.

At Abu Zenima, in Western Sinai, oil impregnations overlie a lignite vein in beds of Cenomanian age (see "Preliminary General Report on the Occurrences of Petroleum in Western Sinai, by Hume, Madgwick, Moon and Sadek).

The geological evidence seems, therefore, to be in favour of a vegetable origin for the Cretaceous oil.

As regards the younger horizons, the succession at Hurghada, from the unconformity upwards, is as follows :—

(*a*) A considerable thickness of marls containing Globigerinael. At the base of this division is a conglomerate in which, and in the associated limestones, oil is found.

(*b*) A lagoonal series, consisting in the main of gypsum with shale and dolomitic limestone interbedded and occasional development of salt. Oil in comparatively small quantities

* *Journ. Inst. Petr. Techn.*, VII., No. 29, *p.* 394, Dec. 1921.

is found at several horizons in the gypsum and its interbedded limestones.

(*c*) Gypseous Shales, Grits, etc. to the surface.

The Egyptian Government recently put down a deep bore in the Abu Shaar area, some ten miles to the N.W. of Hurghada. This bore encountered a considerable thickening of the lagoonal phase, a large mass of salt being pierced. The bore did not reach the Globigerina Marls (assuming them to be here present), but was still in younger beds at 3505 ft. Carbonaceous shales were encountered at several points in this bore, this being the first known occurrence of such shales in Tertiary beds in Egypt.

A second bore put down by the Government in the same area passed through carbonaceous shales within a short distance of the ground surface. In neither bore was actual oil encountered.

Diatoms were found by Mr. F. W. Moon in two bands in the first of these two bores, and he has subsequently found them in many other bores at widely separated points ; but in every case in beds of Miocene, or younger age.

Oil of Miocene age has, in the past, been produced in considerable quantity at Gemsah from a Gypsum-Limestone-Salt series ; this oil is also of very light gravity. No geological evidence as to the probable origin of the oil is to be found by study of these bores.

The suggestive points in connection with the possible origin of these Miocene oils (which are entirely different from the Cretaceous oils) are :—

(*a*) The development of considerable thicknesses of marls with Globigerinae. (In places these marls contain also fish scales.)

(*b*) The evidence recently obtained by Mr. Moon as to the widespread occurrence of Diatoms in the oil fields region.

(*c*) The recent evidence of the Abu Shaar bores as to the existence of Carbonaceous shales in the younger beds.

No definite conclusion seems possible on this evidence at the moment.

Mr. F. W. Moon also wrote :—With regard to the paragraph beginning " It would be interesting if some supporter of the animal theory would conduct . . . experiments, etc.," assuming that some petroleums have originated during the processes of accumulation and decay, both of animal and vegetable matter, under ancient lagoonal and deltaic conditions, I have long thought that the time has come when a thoroughly equipped expedition to the many countries, tropical and semi-tropical, where large lagoons occur in which animal life abounds, where teeming millions annually live and die, and where vegetable matter grows and is deposited in great abundance, to study present-day lagoon conditions generally,

and to make collections, and analyses for hydro-carbon content, of lagoon waters, muds and deposits from various depths.

Fresh—and salt-water lagoons, intermittent lagoons, large deltas, and even deposits from shallow seas, could all be included, and made the subject of real investigation, so that a thoroughly reliable report on the origin and occurrence of hydro-carbons could be placed in the hands of the Petroleum world, to clear up, if possible, our doubts about the mode of origin of petroleums which we now find in strata called "lagoonal" or "deltaic" deposits.

In reply, **Mr. Cunningham-Craig** wrote:—Mr. Moon's idea is an excellent one; there is a great deal to be learnt by studying accumulations that are forming at the present day under lagoon conditions, either in fresh or salt water. However, much work of this kind has already been done, and oils in small quantities have been made from sludge accumulated in harbours and lagoons, but it is found that if the sludge be excavated to a depth of two or three feet the organic contents have decreased enormously. This applies not only to the animal matter, but often to vegetable matter; the former almost completely disappears, and the latter also decomposes to some extent. Thus, in a thick deposit it is only the upper layers that are rich in organic matters, except, of course, where there is a quantity of heavy tropical timbers sunk and buried in the lagoon deposit.

The International Petroleum Exposition and Congress, Tulsa, Oklahoma, U.S.A., October 8th to 14th, 1923.

By F. B. PLUMMER, M.Am.Inst.Min.Eng., M.Am.Assoc.Pet.Geol.

A FIRST impression of the International Petroleum Exposition at Tulsa, Oklahoma, was that it was going to be an ordinary Oklahoma Fair, but this unfavourable impression was soon dispelled as row after row of petroleum exhibits arranged in stalls along each side of roofed passageways came into view. The rows of booths were laid out in parallel alignment completely covering two city blocks. Wandering among splendid examples of modern drilling equipment, latest types of engines, huge compressors, pumps, tanks, refinery equipment, and in fact representations of every branch of the petroleum industry, the premature conceptions of a country fair soon gave way to real satisfaction and admiration for a great exhibition. Most of the machinery was set up completely equipped and actually in operation exactly as if at work in the oil-fields. There was the opportunity to study all phases of oil-field, as well as refining, operations in one condensed space.

The first impressions that the exhibits were local and not representative of foreign workmanship or worthy of the name "International" persisted, however. It was not an international exposition. All the displays were American, and by far the greater number were furnished by Mid-Continent and Oklahoma companies and dealers. It would have been so much more instructive and so much more complete if equipment of European and British manufacture could have been added, with the new British inventions placed side by side with the American. The location of the Exposition so far inland, of course, hindered foreign participation, and it is to be hoped that at some time a similar exhibition may be held in London or New York, so that both great oil-producing nations can be fully represented. Then and then only can it be really an international display.

It would be impossible to describe all the exhibits at the Exposition without writing a treatise on the oil industry. Only two or three which received the most attention from visitors will be mentioned, and it is hoped that these descriptions may serve as examples in presenting some idea of the whole.

Portable drilling rigs.—Light portable drilling rigs, both of the rotary and standard types, are becoming more and more popular

or drilling in fields where the oil strata are shallow. These machines are also being adapted for geological exploration work in salt-dome

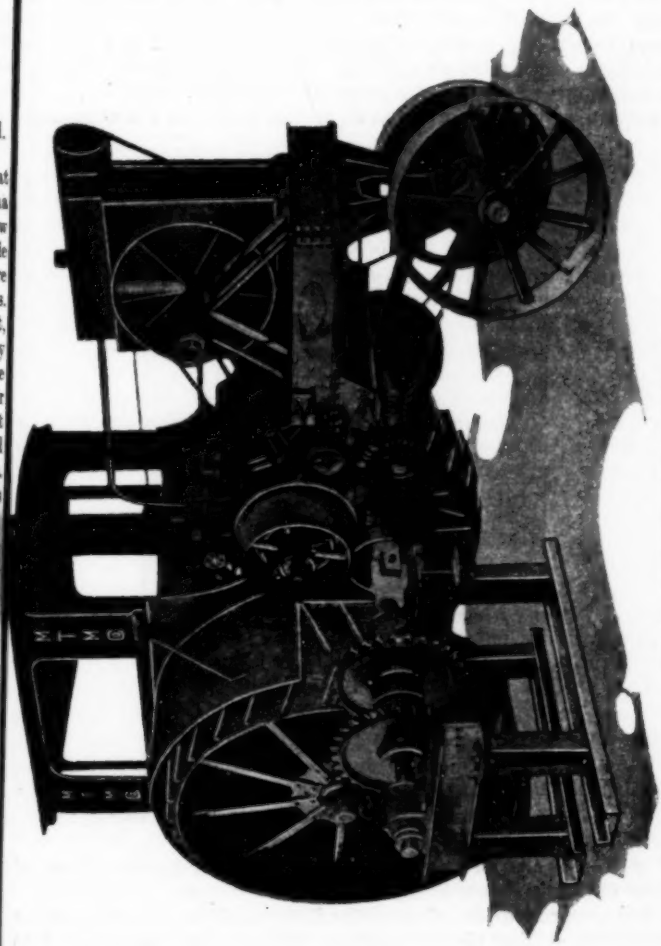

FIG. 1. PORTABLE DEEP DRILLING MACHINE.

fields and other areas where the subsurface structure can be ascertained only by core drilling. Several types of light portable rigs were shown at the exposition, and it was demonstrated that, by

using a tractor driven by a gasoline or kerosene engine, and equipped with a special kind of clutch to drive a special light rotary rig, test holes could be drilled to one thousand feet or less for one dollar per foot. It was shown also that under favourable conditions even much deeper holes could be put down with this outfit and oil wells brought in much more cheaply than by the old methods. The rig, including a steel derrick, was so constructed that it could be readily transported by means of the same tractor engine to a new location

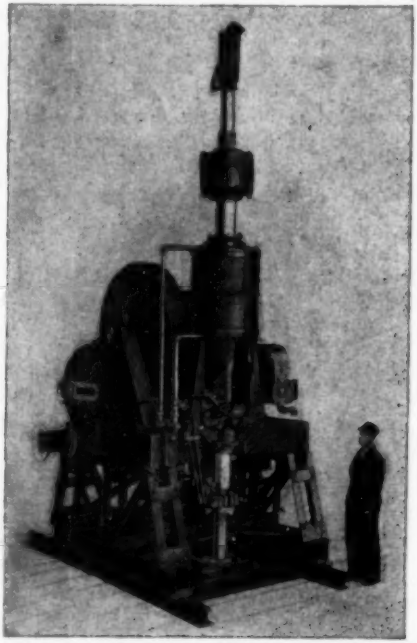

FIG. 2. HEAVY DIAMOND CORE DRILL, FRONT VIEW.

at minimum cost of time and expense. An illustration of one of these outfits is shown in Fig. 1.

Diamond core drills.—One of the features of the exposition was a giant core drilling rig in operation. This devise was designed to bore an eight-inch hole for oil, set casing, and bring in oil wells to depths of 3,000 feet in shorter drilling time than by the old methods. The process offered the great advantage also of taking a complete core all the way down. The new outfit appeared to many to be a

real advance in drilling methods. Several technical men predicted that it would come into universal use in certain oil fields. Illustrations of the core drill are shown in Figs. 2 and 3.

Geological exhibits.—The United States Geological Survey and Bureau of Mines exhibited interesting models of several American oil-fields and displayed a large number of new oil-field maps and sections. One exhibit that aroused a great deal of interest was a

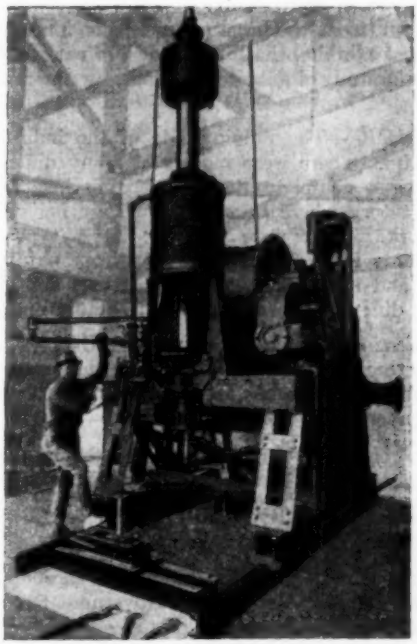

FIG. 3. DIAMOND CORE DRILL, SHOWING DRILL RETRACTED FROM HOLE, READY TO HOIST THE RODS.

large circular cardboard disc, about three feet in diameter, marked off into sectors about one inch wide at the circumference of the circle. This disk was fastened so that its edge rotated beneath a high-power microscope. At the end of each segment were placed glass slides on each of which a group of foraminifera and other minute fossils actually collected from well cuttings was mounted carefully. Each group characterised some well-known geologic

horizon in the Mid-Continent oil-fields, and the slides were arranged in stratigraphic order. By rotating the disk one could bring the minute and little-known fossils of each successive horizon, or any given horizon, beneath one's view and study them by means of the microscope. It was explained that this scheme made it very easy to compare quickly an unknown microfauna, washed from a new sand sample obtained in drilling, with many different sets of known characteristic micro-fossils, thus enabling the geologist to recognise his unknown horizon more easily.

It is apparent, from the large collections of foraminifera shown in the geological exhibits, that a great deal of research work is now being done in the study of microscopic fossils by American petroleum geologists.

Refinery Exhibits.—The refining side of the industry was well represented also. There were models of many different types of stills, including a complete Burton cracking installation, and a chemical laboratory where chemists were carrying on oil and gas analytical work. There were also the latest types of gasoline extraction plants for recovering gasoline from natural gas, huge compressors, and models of pumping stations and pipe-lines, as well as many different types of pumps in actual operation.

Technical meetings.—Each day during the Exposition there were technical meetings in which papers were delivered by experts, discussing various phases of the oil industry. Among the more noteworthy papers were the following:—

Prospects of oil in Guatamala. By Senor Don Francisco Latour, Minister from Guatamala at Washington.

Petroleum in Peru. By Senor Santiago F. Bedoya, Secretary of the Peruvian Embassy at Washington.

Oil Industry and Petroleum Laws in Venezuela. By Senor Lucio Baldo, Representative of the Government of Venezuela to the Exposition.

Oil in Ecuador. By Senor J. Cueva Garcia, Official Delegate from Ecuador.

Nationalization of Petroleum in Rumania. By George Anagnostache, Delegate from Rumania.

China's Interest in Oil. By T. O. Chu, Member of Geological Survey of China.

The members of the Petroleum Division of the American Institute of Mining Engineers who were present at the Exposition, held an interesting meeting, at which all the chief processes for separating

oil from emulsions were discussed. The following papers were the most noteworthy of those delivered :—

Principles of Chemical Treatment of Cut Oils. By E. O. Sanguinet.

Electrical Treatment of Cut Oils. By F. D. Mahone.

Separation and Purifications of Liquids by Centrifugation with Special Reference to Petroleum. By A. F. Meston.

Altogether the Exposition was extremely interesting and instructive. In viewing the 175 or more exhibits one could have a complete survey of the American oil industry within a comparatively short time. Such an inspection would take a much longer time, and probably be much less thorough if conducted in any other manner than that made possible by the International Exposition and Congress. The only regret is that it could not have been truly that which was indicated by its name, International. Especially it is a pity that exhibits illustrating British manufacture and invention could not have been included, and also that more members of the Institution of Petroleum Technologists could not have been present to have contributed to the meetings.

STUDENTS' SECTION.

LONDON BRANCH.

THE inaugural meeting of the London Branch of the Students' Section of the Institution was held at Aldine House, Bedford Street, London, W.C. 2, on Tuesday evening, October 23rd, 1923, and was attended by thirty-six Students and visitors.

The President of the Institution (Mr. H. Barringer) opened the meeting, and said it gave him great pleasure to welcome the Students present, on behalf of the Council, and to wish them every success. He believed that the date would stand in the annals of the Institution as marking a new development. The Institution looked to its Students to carry on and advance its causes, and, although they must be independent and use their own resources, all must pull together for their mutual benefit.

Mr. Arthur W. Eastlake (Vice-President) seconded the President's remarks, and said that the Section must row its own boat and make of itself a success. Knowing many of the Students as he did, he was sure this would be done.

Mr. Ashley Carter (Member of Council) wished the Section prosperity and success.

Mr. H. G. Austin (Chairman of the London Branch) then took the chair, and expressed indebtedness to the President, Members of Council, and the Secretary for their presence. He referred briefly to the formation of the Section and the arrangements which had been made for its meetings, and hoped that as the Section became more widely known, a larger number of members of the Institution would attend its meetings. He then called on Mr. F. R. S. Henson to read his paper, an abstract of which follows :—

THE USE OF THE HAND AUGER IN GEOLOGICAL MAPPING.

By F. R. S. HENSON (Student Member).

Geological Mapping.—In preparing a report, the geologist has two main tasks before him : First, to examine the strata and determine the nature of the rocks and their position with regard to each other ; secondly, to unravel the structure—that is, the

disposition of the strata and the nature of the folding or other distortion of the beds.

The strata may remain horizontal, as originally deposited, or they may be flexed or folded into anticlines and synclines. Complex folding on a smaller scale is frequently met with, giving rise to steep local dips, while faulting is a common occurrence.

The continuity of the strata may be broken by local denudation, causing it to outcrop at the surface of the ground, and it is upon these outcrops that the geologist relies for his evidence.

Junctions between the outcrop of one stratum and those of the strata above and below are followed and plotted in projection on a topographical map, and the boundary lines limiting these outcrops accurately fixed.

The geologist first endeavours to form some idea of the general dip and strike of the rocks and, working systematically over his map, examins every exposure of rock, and every detail, however small, that may indicate to him the nature of the rock outcropping at each point, recording his observations on his map. In his search he must examine all excavations of any kind in the hope that rocks may be exposed beneath the soil.

The soil and vegetation themselves will often yield evidence. Rocks, on disintegrating, may yield distinctive solids which indicate the type of rock beneath, and different soils favour different types of vegetation.

Wherever a good exposure of solid rock exists, both the amount and direction of its dip should be taken. Care is needed in taking dips, as no dip is a *true* dip unless it is measured in a direction at right angles to the strike, and because soil creep, etc., will often disturb strata outcropping at the surface.

The geologist's map should now be covered with information, and he should be thoroughly familiar with the area under consideration. He can now fill in the boundaries between successive strata.

Determination of Structure.—From the foregoing notes it may seem that the geologist has no idea of the structure until he has put in his boundaries, but the interpretation of structure is only a matter of clear thinking in terms of three dimensions, combined with experience.

The recording of accurate dips, etc., will admit of treatment of structure on a mathematical basis. Given the dip of a bed, its thickness, and the slope of the ground, the breadth of its outcrop can be calculated. By mechanical means the structure may be

obtained by drawing a vertical section to scale, preferably in the direction of dip. The topographical profile may be drawn to a vertical scale from the contours, and the position of junction lines taken from the map, the underground disposition of the strata being made to fit the position of the outcrops and other evidence.

From a geological map, anyone familiar with map-reading should be able to decipher the structure of the area on common-sense theoretical principles—*i.e.*, the intimate relations between structure, topography, and the course of outcrops. Of these, structure is the primary factor, while topography is developed later on and the nature of that development depends largely upon the pre-existing geological structure of the area, and upon the lithology of the rocks. The course of the junction lines is entirely dependent upon the first two factors of structure and topography. Given two of these factors, we may deduce the third, but it is usually structure we are asked to find.

Auger Drilling.—The hand auger used by the author is a half-inch wood auger, with a thread turned on the stem to connect an adapter, which in turn screws on to a suitable length of gas piping. This piping is threaded at each end so that the upper end may be connected through a union to another length of pipe or to a T-piece, the auger being turned by a tommy bar through the T-piece. Two footprints, for making and breaking joints, a water bucket, and a long iron spike for smashing through gravel patches and other obstacles, complete the equipment.

In operation the auger is forced into the ground by turning the tommy bar, weight being brought to bear upon the latter, and at every few turns the bit is jerked upwards to prevent it penetrating too deep. After about every half-a-dozen turns the bit is removed from the hole, and a good core of the material penetrated will be found in the channels of the bit. While drilling, the hole should be kept full of water in order to lighten the work of turning the auger and to ensure a good core being obtained. With such an auger it is possible to drill a hole to a depth of 15 feet or more in soft rocks. The use of the auger has two main purposes :—

1. As a means of bringing up samples of the undisturbed beds beneath the surface.
2. As a means for stratum contouring and for examining minor structural features.

Stratum contours are merely strike lines plotted at regular vertical intervals, and to prepare these we must find the height above Ordnance Datum of our junction at a number of chosen points. To contour at regular intervals, we may interpolate

between these points, assuming the inclination to be constant over a small area.

Assuming that we wish to determine the outcrop of a junction between a clay on top and a sand below, giving a topography from which it is very difficult to find the boundary by sight. A hole is drilled straight past the junction, into the sand beneath, and a characteristic horizon selected as a key for the registration of depth. The boundary is run in roughly by sight, and sites for holes selected at distances of from 50 to 200 yards along the boundary and some 5 feet vertical above it, alternate holes being staggered by carrying them 50 yards back. The position of each hole is marked, and the level of the top of the hole determined.

The rest of the work may now be done in the drawing office. It is easy to interpolate between the stratum levels obtained with the auger, and the junction plane may be contoured in 5 feet vertical intervals and the boundary plotted in.

By this means it is possible to draw rough conclusions as to minor folding, etc., in a series of beds, even though these folds may be concealed hundreds of feet below the ground. This is done by augering along a number of successive boundaries and detecting minor folding, etc., immediately above each junction. These being established, it is probable that the folds will be present in the underlying beds as far down as the conformity of the strata holds.

The paper concluded with a short description of the use of the hand auger in Texas and Trinidad, and was illustrated throughout with a large number of excellent lantern slides.

Mr. H. G. Austin said he thought that all present would agree that the paper had been of great interest, and that the slides had brought forward many points which might otherwise have been missed.

Mr. A. Nutsford asked to what type of rocks the hand auger was most adapted, or was it only suitable for junctions between soft rocks.

Mr. C. A. Sansom asked how auger holes could be spaced along a steep slope representing the junction between a clay and a sand, since it appeared that very deep holes would be required to penetrate the surface material and reach the junction.

Mr. A. B. Miskin asked if the author could suggest a method of getting through gravel pockets, which in his experience has caused the discontinuance of work in many cases.

Mr. A. Nutsford suggested that the use of water in the hole when drilling caused the side of the hole to fall in, and that better cores could be obtained with a dry hole.

Mr. A. L. Wilson said that when drilling through sand into clay, with a wet hole, the sand was likely to fall in and contaminate the clay, giving the impression that one was again drilling into sand.

Mr. A. G. G. Hanks said that when drilling through a sand and clay junction it was possible to feel the approach of the junction by the grating of the bit on the sand.

Mr. H. G. Austin asked what instrument was used for fishing if the auger became broken.

Mr. Henson, in reply, said that the ordinary wood auger could be used to a certain point, but the type used in Texas was more suitable for the harder rocks. The selection of sites for holes on a steep slope did offer difficulties, but it was unusual to find a slope wholly composed of clay, which generally appeared only as a veneer at the top. With regard to gravel pockets, it was possible to drill through these by substituting an iron spike for the auger.

Judging junction by feel was only a rough guide, and for greater accuracy the core must be examined for the presence of glauconite, etc. In the event of a bit being lost in the hole, a brass socket was lowered over the broken thread, and a thread cut in the socket, thus securing the bit.

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Abstracts.

Compiled by S. Bowman, A.I.C., P. Dvorkowitz, Ph.D., H. Moore, M.Sc., A.I.C., F.C.S., F. G. P. Remfry, B.A., D.Sc., A.I.C., and T. A. Smith, B.Sc., F.I.C.

General.

The Composition of Paraffin Wax. Part 3.—F. Francis, G. M. Watkins & R. W. Wallington. *J. Chem. Soc.*, 1922, 2804–10.

In part 2 (*J. Chem. Soc.*, 1922, 1529–35) the authors described the isolation by means of repeated fractionation under reduced pressure, of seven fractions of constant boiling point, constituting 80% of the wax under examination.

The constants of these fractions were as follows :—

Fraction.	B. Pt. 0.05 mm.	D 100/4.	M.P.	Mol. Wt.
B	150°–151°	0·7453	44·9°	325
C	160·8°–161·3°	·7501	51·9°	332
D	169°–170°	·7523	53·2°	358
E	180°–182°	·7555	57·3°	394
F	190·5–191°	·7596	60·9°	419
G	196°–198°	·7630	63·3°	449
H	217°–219°	.7678	66·6°	434

In the present paper an attempt is made to prove that these fractions are pure hydrocarbons, and while this is not conclusive strong evidence is advanced to show that they are not mixtures. Each of these fractions are oxidised at 100° C. with air in the manner described in Part I. (*J. Soc. Chem.* 1922, 496), and although turpentine was employed this was found to be unnecessary, as the effect of prolonged heating during fractionation was to considerably diminish the latent period of oxidation.

Separation of the various products of oxidation was carried out by treating the alcoholic solution with potash, extracting unoxidised material with petroleum ether, and liberating the acids from their salts.

The percentage yield, M.P., and acid value of the products obtained from each of the seven fractions is given, and the fact that the physical constants of the unoxidised material were identical with the original fraction in all seven cases is regarded as more than coincidence, and is strong indication that each of these fractions was a pure hydrocarbon.

S. B.

Explosions in Liquid Air Rectification Plant.—E. Fyleman. *J. Soc. Chem. Ind.* **42**, 4, 57–39.

The paper describes experiments made to ascertain the cause of often violent explosions which occur in liquid air rectification plants during

the manufacture of oxygen and nitrogen. Although these have generally been attributed to the presence of acetylene, and in fact solid acetylene does explode when stirred with liquid oxygen in a copper vessel, elaborate purification of the ingoing air from this gas has not eliminated the possibility of these explosions. In his preliminary experiments the author confirmed the fact that commercial oxygen as well as liquid oxygen and liquid air, contain small amounts of organic matter, by passing purified air first through soda lime and then over red-hot copper oxide, and finally through baryta solution. This is shown to be due to the slight vapour pressure of the lubricating oil used in the compressor. Various experimental conditions were produced in order to test the effect of the presence of this organic matter, and also that of ozone and superheating of the liquid gases, but without result.

Reverting to the idea of acetylene the author found that when oxygen was passed through heated compressor lubricating oil, and the exit gases passed through asbestos wool, soda lime, and a 5% ammoniacal solution of silver nitrate, respectively, the latter solution began to be reduced when the temperature of the oil reached 240° C. At 315° C. explosions took place, and a gas resembling acetylene was evolved forming a yellow silver salt, which on washing and drying, detonated violently on being gently stirred.

The above experiment is considered dangerous and demands extreme caution. Using air instead of oxygen a temperature of 440° C. was reached without any explosion, although the oil itself began to distil over with decomposition. If, however, the air be used at a pressure of 30 atmospheres acetylene is formed at a temperature of 350° C. The chemical nature of the oil appears to be immaterial, ordinary paraffin wax and olive oil giving the same results at only slightly different temperatures. These results show how acetylene gets into the plant, and the obvious necessity of keeping all the lubricated parts of the compressor cool. By taking such precautions all tendency to explosions can be avoided. S. B.

THERMAL DECOMPOSITION OF PETROLEUM RESIDUES AT REDUCED PRESSURE.—J. Reilly and E. W. Blair. *J. Soc. Chem. Ind.* **41**, 302–3T (1922).

American petroleum residue, obtained by distilling crude oil at low pressures with superheated steam, has been fractionally distilled in glass and copper retorts. The residue contained C. 87%, 1·13% had a flash point of 238° and a density of ·883. The unsaturation value was equivalent to 85 mg. of Br_2 per grm. of petroleum. The distillations were carried out under pressures ranging from 1 atm. to 1 mm. As the pressure was reduced there was a decrease in the oil volatile in steam, and in the degree of unsaturation of the distillate as a whole. A partial separation of the saturated and unsaturated portions of the distillate can be effected by steam distillation. T. A. S.

DIMETHYLCYCLOHEXANES.—G. Chavanne and P. Becker. *Bull. Soc. Chim. Belg.* **31**, 95–98 (1922).

When xylenes are reduced in the presence of Ni or Pt. catalysts, products of different physical constants are obtained. It is stated that the substances derived from *m*- and *p*-xylene in the presence of Pt. are mixtures and the purity of the *o*-xylene reduction product is doubtful.

The product obtained from *o*-xylene in the presence of Pt. is different from that obtained in the presence of Ni. Cis and trans-stereoisomerism is the suggested explanation. T. A. S.

GUM-FORMING CONSTITUENTS IN GASOLINE.—N. A. C. Smith and M. B. Cooke. *Amer. Bur. of Mines Rept., No.* 2394 (1922).

Little is known of the nature, origin, and formation of the gumming substances deposited when certain gasolines are evaporated. During the war it was specified that aviation gasoline should be free from these substances as determined by the Cu-dish test. Examination of the 850 samples of gasoline collected for the Bureau's gasoline survey of 1919, showed that the Cu-dish test was incompletely standardised and that many factors contributed to the unsatisfactory results obtained. In one case the gumming residue obtained in the dish was light in colour, but in every other case the residue was jet black. As this might be due to Cu S glass dishes were substituted in some of the preliminary tests. A sample of gasoline which gave 0·18 gr. of residue when heated in a Cu-dish gave 0·011 gr. when evaporated rapidly from a pyrex glass beaker, but 0·1 when evaporated slowly from glass. A sample heated in a current of air in an Erlenmeyer flask fitted with a reflux condenser for several days gave 4 gr. of gum. The presence of water in a sample treated similarly caused gum to deposit during the heating, but the quantity of gum was no greater. By bubbling hydrogen through a sample only 0·1 as much as gum was obtained as was produced by bubbling air. No direct connection appeared to exist between gum formation and the unsaturated hydrocarbon content of the gasoline as determined by absorbing the unsaturated hydrocarbons in sulphuric acid. A gasoline containing 18% of unsaturated hydrocarbons was heated for 100 hours in the presence of water and abundance of air. After steam distillation of the volatile hydrocarbons the gum was determined. After several repetitions of this process the total gum obtained from 100 c.c. of gasoline was 7 grms. The recovered gasoline still contained 17% of unsaturated hydrocarbons. Two methods to replace the Cu-dish test were developed but were considered too involved for general use These depended upon steam and vacuum distillation of the gasoline. The method finally adopted was as follows : 20 c.c.m. of gasoline are evaporated from a 30 c.c.m. glass evaporating dish by heating for 4 hrs. on an actively boiling water bath. The residue is dried for 24 hrs. at 105° C. The gum is soluble in acetone when freshly formed, but becomes partly insoluble on prolonged heating. Formaldehyde-phenol condensation products are suggested by this behaviour, and also by the ultimate analysis of the gum. Tests for aldehydes in gasoline were obtained by the ammoniacal silver nitrate and the Hehner methods ; but as only negative tests for phenol were obtained the possibility of phenolformaldehyde condensation products was dismissed. Aldehydes alone gave similar resins when evaporated alone, or in solution in gasolines in the presence of a trace of sulphuric acid. Treatment of aqueous extracts of cracked gasolines with bisulphite gave crystals which, on microscopic examination, were found to bear resemblance to those obtained from valeraldehyde. The gum-number of a gasoline can be reduced by washing with water. The formation of aldehydes by oxidation of the unsaturated hydrocarbons is considered a probable explanation of the presence of gumming substances in gasoline. Long-time storage tests on a number of gasolines are in progress. Tests will be made every 60 days, and the influence of air, water and iron (cylinders) are being considered. T. A. S.

ALCOHOL FROM TRENT AMALGAMS.—*Chemical Age*, Vol. VIII. No. 186, 1-2.

Coke-oven gas, containing 3% of ethylene, treated at Skinningrove for the recovery of alcohol yielded over 1½ gals. of alcohol per ton of coal. The question of the recovery of alcohol from the gases produced by the distillation of the Trent coal-oil amalgams has received attention in America by J. D. Davis, and gas very rich in ethylene has been obtained by feeding the amalgam into retorts kept at suitable temperatures. At 500° C. a gas containing 17% of ethylene was obtained, whilst at 400° C. the yield of ethylene was 23·4%. From this gas it should be possible to recover 30–32 gals. of alcohol per ton of amalgam distilled. The loss of calorific power of the Trent gas due to the removal of the ethylene is approximately 15%. T. A. S.

CHLORINATING PARAFFIN HYDROCARBONS.—G. T. Koch, Assr. to Ohio Fuel Supply Co.

Liquid hydrocarbons of the paraffin series react with chlorine gas under the influence of actinic rays, and in the presence of an absorbent such as silica gel, activated carbon, or finely-divided pumice. By passing the hydrocarbon and chlorine through successive quantities of the absorbent the reaction can be made to proceed more quickly and smoothly, and the resulting production of chlorohydrocarbons can be made more definite in character and amount. The reaction can also be hastened and improved by impregnating the finely-divided absorbent with metals or metallic compounds. T. A. S.

THE GENESIS OF PETROLEUMS.—H. Hofer. *Pet. Times* **8**, 204–827.

It is suggested that mineral oils be classified as: (1) methane oils; (2) naphthene oils, and (3) naphthene-methane oils. The first class would be those containing not less than 60% of methane or paraffin hydrocarbons, the second a similar percentage of naphthene or asphaltic oils, and the third approximately equal quantities of the first two. A fourth group has been proposed for those oils containing a large proportion of hydrocarbons other than those of the methane or naphthene series. In the United States oils are generally classified as those with a paraffin or asphaltic base, the latter being characterised by the deeper colour. From a careful study of analyses of European and Asiatic oils Hofer has concluded that oils with an asphaltic base, *e.g.*, the Baku oils, belong to a younger geological formation, and that in the Carpathians at any rate, the older the formation from which the oil is derived, and the greater the tectonic movements, the higher is the quality of the oil. The age limit of the naphthene oils is not greater than rocks subsequent to the Paleozoic age. Oils obtained from older strata are either of mixed type, or have a paraffin base. In America asphaltic base oils are found in California in Tertiary formations, in Texas and Louisiana in the Upper Chalk. The Lower Carbonaceous in Pennsylvania and West Virginia yields methane base oils, and in the Ohio for mid-continent fields mixed oils are obtained from the Upper Carbonaceous. Paraffin base oils are obtained from strata which have been subjected to considerable earth movement, and in which metamorphism has more or less occurred. In the Mid-Continent and Ohio fields faulting and metamorphism is much less than in the Pennsylvania field. From these facts Hofer concludes that the naphthene oils are younger than the methane oils, and the mixed oils lie chronologically between the two and that geotectonic conditions are largely responsible for the quality of the oil. The evidence seems to show that, assum-

ing oils to have originated from altered organic matter such as fats, fatty acids, etc., methane oils are modifications of naphthene oils. Hofer terms this change 'methanisierung,' and it is obviously not complete in the case of mixed oils. The history of the oils seems to be: (1) Action of enzymes and bacteria on the mother material; (2) the naphthene oils; (3) tectonic conditions resulting in the formation of methane oils. In cases where there has been more active faulting and metamorphism the methane oil has been converted into natural gas. In Burma, Java and the East Indies the oil found in younger rocks may contain as much as 40–50% of methane oil. This indicates more earth movement than in the Californian fields. T. A. S.

THE HIGH-TEMPERATURE TREATMENT OF HYDROCARBONS AND PHENOLS.—W. Allner. *Petroleum*, 1922, **18**, 1337–1350.

I. Roumanian petroleum pitch was distilled in gas works retorts to prepare electrode coke. The coke did not adhere to the walls of the retort when the temperature was not lower than 1000° C. Excessive cracking and choking of the pipes with carbon took place when steam was not used to facilitate the process. With steam distillation the yield of petroleum coke was 15·9%, and the optimum temperature 1100° C. The ash content of the coke was 1·5%. The heavy hydrocarbon content of the gas given off is greatest immediately after charging, and at the lowest working temperature of 1065° C. The hydrocarbon content of the gases when steam was used was, at its maximum, 38·8%. The gas, mixed with the gas from coal-fed retorts, considerably raised the heat value of the mixture. The oils condensed from the gas from retorts without steam, when fractionated, underwent cracking decomposition at 250° C. The oils from steam distillation distilled well, giving a yield of 77% up to 370° C. By filtration of the original residues the ash content of the coke was reduced to 0·25%. The hydrocarbons were removed from the gas by scrubbing with a petroleum distillate of 0·927–30 sp. gr. The production of hydrocarbons is greatest about half-an-hour after charging. In total, about 70 g. per cb. m. of light oils were recoverable from the gases, or 2½ to 3% on the petroleum pitch employed. The production of electrode coke is only economically feasible when the gases and light hydrocarbons are also utilised. The process cannot be economically worked in fire-clay retorts. H. M.

THE HIGH-TEMPERATURE TREATMENT OF HYDROCARBONS AND PHENOLS.—W. Allner. *Petroleum*, 1922, **18**, 1369–1378.

II. Experiments were made on the cracking of petroleum oils in gas retorts, with a view to the production of fuel for internal-combustion engines. The oil was blown into the retort by a Körting injector along with steam. The gases were washed with a petroleum distillate of 0·881 sp. gr. At a temperature of 1250° C. the gases produced contained 30·6% of hydrocarbons, 30·2% of methane, and had a gross heat value of 9875 cal. per cb. m. The total products from 100 kgr. of petroleum oil were 28·4 cb. m. of gas, 12·5 kgr. of thick tar from the receiver, 59·2 kgr. of tar from the pipes, of which 35·2 kgr. were a petroleum fraction distilling up to 300° C., and 2·1 kgr. of light oil recovered by scrubbing. Gas tar oil was sprayed into a retort half-filled with coke, with a view to obtaining light motor oils. The resultant gas contained 8.8% of hydro-

carbons, 35·4% of hydrogen and 31·6% of nitrogen, and had a gross heat value of 5700 cal. per cb. m. The gases were scrubbed with oil, and 100 kgr. of tar gave 21·4 cb. m. of gases, 17·8 kgr. of tar, and 2·5 kgr. of oils obtained by scrubbing. An analysis of the recovered light oils from petroleum pitch distillation showed the presence of benzene, xylol and naphthalene, formed by the heat treatment from aliphatic compounds. The light oils had a composition as follows: paraffins, 12·6%; olefines, 51·0%; and aromatic hydrocarbons, 36·5%. Light oils from the petroleum cracking process contained benzene and toluene, but no naphthalene. They contained paraffins, 6·2%, olefines, 35·6%; and aromatic hydrocarbons, 54·2%. In the light oils from the cracking of tar, benzene, toluene, the three xylols and small quantities of naphthalene were found. Their analysis showed paraffins, 5·3%; olefines, 21·0%; and aromatic hydrocarbons, 73·5%. Experiments were made in an electric tube furnace, and the optimum temperature for the treatment of tar oil was found to be between 550° C. and 600° C. H. M.

THE SIGNIFICANCE OF SURFACE TENSION IN NAPHTHOLOGY.—L. Gurwitsch. *Petroleum*, 1922, **18**, 1269–1278.

The surface tension of a liquid in air or another immiscible liquid is determined by measurement of the size of the drop. If a liquid issues in drops from a capillary having a radius of opening r, σ being the surface tension in air, the drop will cling to the capillary with a force $= 2\pi r\sigma$, and will only be detached when its weight p exceeds this value. In estimating the surface tension in water the difference of the weight of the drop against water is taken as p. Dropping must take place slowly, and the drop should not adhere to the glass section of the capillary. The drops of an emulsion tend to unite by virtue of the surface tension of the liquids in contact. If a solid substance is in solution in one liquid the surface tension is decreased and demulsification hindered. The asphaltic substances in crude petroleum give consistence to the film which separates the drops of the liquid, and tend to concentrate in that film. A 1% solution of resinous substances in various media, as spindle oil and paraffin oil, gives films of different resisting powers, corresponding to the lowering of the surface tension of the solvent. A film may be formed by the presence of petroleum soaps. Emulsions of honey-comb structure are persistent. The Bibi-Eybat emulsions are formed by petroleum soaps, and are resolved by the addition of ½% of naphthenic acids. Surface tension affects the formation of emulsions in which solid substances, such as fine sand and mud, are present. Very persistent emulsions, not resolved by naphthenic acids, are thus formed. Soap emulsions may be formed by the alkali washing of petroleum distillates, their persistency varying with the nature of the solvent. Sulphonic acids greatly lower the surface tension, and lead to the formation of persistent emulsions. The formation of emulsions from alkali washes depends upon the temperature of the operation and the concentration of the alkali. The addition of alcohol diminishes the tendency of alkali to form emulsions. The fitness of an oil for turbine lubrication may be judged by the determination of its surface tension, as also the fitness of an oil for the lubrication of the cold-running parts of marine engines, for which emulsifying properties are an advantage, and of steam cylinder oils and cutting oils, etc. H. M.

SOME NEW NITROGEN-CONTAINING HYDROCARBONS FROM BAKU PETROLEUM. —E. Pyhälä. *Chem. Zeit.*, 1922, **46**, 953.

The tarry constituents of the kerosene acid sludge from Baku petroleum were precipitated by water and, after standing, distilled with superheated steam. The distillate contained benzene, toluene, hydrocarbons with an odour of terpenes and naphthenic acids. It was shaken up with sulphuric acid and neutralised with strong solution of caustic soda, the temperature rising and gases being given off, and small quantities of oil and water distilling over. The gases contained ammonia. The distillate was a pale yellow oil, and on fractionating the greater part came over between 75° and 80° C. All the fractions gave precipitates with platinum chloride, soluble in chloroform, and of brown, red or violet colour. The presence of amines was indicated, evidently not pre-existent in the crude oil, but formed during its treatment. These may have been formed from amides of the organic acids contained in petroleum, and the amides may have been formed by the reaction of organic acids and ammonia, as salts of ammonia are sometimes present in the strata of the oil fields. H. M.

THE MECHANISM OF THERMAL DECOMPOSITION OF THE PENTANES.—G. Colingaert. *J. Amer. Chem. Soc.* **45**, 130–135.

The object of this investigation was the possible utilisation of the lower boiling fractions occurring in petroleum by means of subjecting n. and iso-pentane to a decomposing temperature and subsequent treatment of the unsaturated hydrocarbons formed to produce acids and alcohols of higher commercial value. In carrying out his experiments the author realised that the products of such decomposition are generally the final result of successive reactions all tending to an increase in the percentage of H formed. In order then to limit the reaction to a primary decomposition the lowest temperature and rate of flow was employed that would ensure a reasonable rate of reaction.

Normal and iso-pentane were each passed through a combustion tube heated to 600° C. at a rate of ½ gram mol. per hour. In this manner 30% of the pentane was decomposed. The total yield of unsaturated hydrocarbons was determined by absorption of the products in bromine water, and the isolation of the constituents by fractionation and crystallisation of their brom-derivatives.

The saturated gases passing through the bromine were condensed by means of liquid air, fractionated and the fractions analysed by explosion with oxygen. In the case of normal pentane 44% of unsaturated, and 56% of saturated hydrocarbons were produced, comprising :—

% Saturated.		% Unsaturated.	
H_2	5		
CH_4	12		
C_2H_6	26	C_2H_4	10
C_3H_8	10	C_3H_6	24
C_4H_{10}	Nil	C_4H_8	3
C_5H_{12}	12	C_4H_6	6

These results correspond to a break in the molecule at the central carbon atom, giving an ethyl and a propyl group, one of which is then saturated at the expense of the other giving : (*a*) ethane and propylene,

and (*b*) ethylene and propane. 55% follows reaction (*a*), and 25% follows (*b*), the remaining 20% consisting of methyl and butyl groups, giving methane and butylene.

With iso-pentane 40% unsaturated hydrocarbons were produced :—

% Saturated.		% Unsaturated.	
H_2	6	C_2H_6	15
CH_4	30	$n.C_4H_8$	20
C_3H_8	24	C_5H_{10}	4
C_5H_{12}	1	C_4H_8, C_5H_8	1

In this case the molecule breaks on one side or the other of the tertiary carbon atom, giving in the first reaction a methane and iso-butane group, and in the second an ethyl and iso-propyl group, these reactions constituting 90% of decomposition. Although *n*-pentane, on decomposition, gives *n*-propyl and ethyl groups, and iso-pentane gives iso-propyl and ethyl groups in the first case these groups form a saturated and unsaturated hydrocarbon whereas in the second an unsaturated product only is formed. As higher temperature would lead to the formation of hydrocarbons containing fewer carbon atoms the yields are not considered sufficient to be of practical importance for the preparation of synthetic organic compounds. S. B.

Application of Cracking System to Grosny Petroleum.—*Journal Grosny Petroleum Industry*, 1922, Nov. 7–9, p. 5.

Generally, from Grosny petroleum the following products are produced : 6–8% motor spirit, 8–10% lamp oil, 0·5 heavy oil, and about 75% residue. The residue represents a liquid, little mobile even in summer time, and which is solid at ordinary temperature. Thus it is difficult to use it as fuel in the winter. The problem, therefore, is to turn the residue into a more mobile liquid.

Attempts have been made to adopt cracking processes, but in view of the formation of unsaturated hydrocarbons and gases, the application of the cracking system was found to be impracticable.

It may be possible to apply the cracking system if measures were taken simultaneously to polymerise the unsaturated hydrocarbons formed.

P. D.

Prospective Petroleum Export for 1923.

In view of the interest of a great many of the English shareholders in Russian Petroleum, the following data is given :—

Products for Export :

(1) *Motor Spirit*, maximum 2½ million poods (about 15 million gallons) at £17 sterling per ton, f.o.b. Novorossisk.

(2) *Kerosene*. On the 1st October, 1922, the stock was 27 million poods. The expected production represents 33·6 million poods. The total consumption for Russia does not exceed 21 million poods. It is expected that there will be for export, not including the Near East and Egypt, 10 million poods, at £3 5s. per ton, f.o.b. Batoum.

(3) *Lubricating Oils*. The stock of 1st October, 1922, was 13 million poods. The expected production is 10·3 million poods. The interior consumption does not exceed three millions poods per annum ; and it is

expected to release for foreign countries five million poods at 1 rouble, 75 k. per pood, f.o.b. Batoum.

(4) *Solar Oil.* 1·2 million poods per 70 k. per pood, f.o.b. Batoum.
(5) *Crude Oil.* 1·5 million poods of paraffinous Grosny Crude at 42 k.
(6) *Spindle Oil.* Half-million poods at 1 rouble, 50 kopecks.
(7) *Cylinder Oil.* 300,000 poods at 2 roubles.

Thus the total quantity of various petroleum products expected to be exported to Europe in the course of the coming year is 21 million poods. And deducting the freight from Baku to Batoum, and from Grosny to Novorossisk, the net sum realised is expected to be about 18 millions gold roubles.

In order to carry out the proposed export trade the Russian Government is considering a project of entering with a Dutch trading firm, called the International Oil Co-operation, which has already made arrangements for distributing petroleum products in Germany, France, Belgium, and Holland. By this agreement a minimum quantity at a minimum price is guaranteed. The surplus profits will be divided between the Government and that firm. P. D.

YEARLY REVIEW OF GROSNY PETROLEUM INDUSTRY FROM OCTOBER, 1921, TO OCTOBER, 1922.

The total quantity of crude oil produced in the Grosny district was 87,652,881 poods, which were produced; from the old Grosny fields, 45,517,649, from the new Grosny fields, 41,956,031; and from the Voznesensk fields, 179,201 poods.

The above quantity was produced:

By baling, 41,718,152 poods.
By spouters, 44,720,325 poods.

Collected from lakes, 1,213,804 poods.

The quantity of the crude oil received was:

Paraffinoid	35,251,359	poods.
Slightly paraffinoid ..	24,859,214	„
Non-paraffinoid	27,542,308	„

On the average in the course of a year, there were 148 baling wells and 11 spouters. Of the quantity of crude produced 10·6% has been used for fuel.

The above data with regard to the ratio of crude produced by spouters to the production by balers, throws a light on the precarious position of the Grosny oil fields at the present moment.

In accordance with a statement published in the Grosny Journal, nearly 45% of the total production in Grosny is produced by two spouters, No. 54 on a plot 147 in the old region, and by No. 2 on a plot 49 in the new region.

In the first, on the 5th December, water appeared to the extent of 15%, and on the 10th of the same month, 20%. Then the daily production which had been 50,000 poods, fell to 20,000 poods.

The total number of workmen employed in the Grosny district Industry is about 10,000, not including the administration and technical staff.

A very interesting table is given, comparing the wages paid pre-war to those paid last year :—

Qualification.	1913–1914 per month	1921–1922 per month
Labourer	24 roubles	5 r. 67 k.
Stoker	35 „	7 r. 68 k.
Baler	30 „	7 r. 79 k.
Joiner	40 „	8 r. 44 k.
1st class fitter	48 „	10 r. 54 k.
1st blacksmith	67 „	10 r. 54 k.

The above table shows that, at present, the workman in the best paid industry of the Soviet government receive wages equivalent to only 16–20% of their pre-war wages. Owing to this nearly 80% of the working population do not receive sufficient wages to provide even their minimum requirements. P. D.

THE MISCIBILITY OF HYDROCARBONS AND LIQUID SULPHUR DIOXIDE.—F. Fontein. *Z. angew. Chem.*, 1923, **36**, 4–6.

The author refers to the paper by E. Zerner, H. Weiss, and H. Opalski (*cf. J.*, 1922, **8**, 546), saying that in relying on the synthetic method the authors named underestimate the danger of error, owing to the presence of small quantities of impurities, and that their statement as to the imperfect miscibility of toluol or tetralin with liquid sulphur dioxide at temperatures below 21° C. is incorrect. Experiments showed miscibility at very low temperatures, but deposition of white crystals. These were due to the presence of water, or of a hydrate of sulphur dioxide. With dry sulphur dioxide the substances were completely miscible at —20° C. The tetralin used must be distilled to free it from oxidised products. H. M.

THE SIGNIFICANCE OF THE TEMPERATURE-VISCOSITY RELATIONSHIP IN THE VALUATION OF OILS.—H. Vogel. *Z. angew. Chem.*, 1922, **35**, 561–563.

The most important of the physical properties of an oil from the point of view of oil technology is the viscosity. It is very desirable that the viscosity should be expressed in absolute units, a desideratum being a simple viscometer that should give readings in absolute units. It is most important that lubricating oils should have as flat as possible a temperature-viscosity curve. Certain oils, the product of a process of electrical polymerisation, possess this quality in a high degree. Experiments were made with a modification of Oswald's viscometer in metal, so made as to exclude the influence of the expansion of the oil with increase in temperature. The indications of this instrument give the value η/s, where $\eta =$ absolute viscosity in centipoises and $s =$ sp. gr. A series of comparative measurements of the viscosities of chemically pure substances allowed a formula to be established for the reduction of the indications of viscometers of the Engler type to absolute units. The The formula gives results agreeing very well with experience for fluids of sp. gr. from 0·6 to 1·5. The viscosity curves may be calculated by the formula $\eta_t = \eta_\infty (t - t_1)/(t - t_\infty)$, where t_1 is the temperature at which the oil is as fluid as water at 20·2° C., and t_∞ is the temperature at which viscosity is infinite and fluidity nil. H. M.

THE REDWOOD VISCOMETER.—W. H. Herschel. *Technologic Paper of the U.S. Bureau of Standards, No.* 210.

Measurements of the dimensions of the Redwood viscometer are compared with those taken by other observers. It was found that kinematic viscosity = $0{\cdot}00260\ t - (1{\cdot}88/t)$ where t is the time of flow in seconds. The error due to inaccuracy in the Meissner formula for average head, $h = h_1 - h_2/\log_e\left(\frac{h_1}{h_2}\right)$, was found to be negligible in ordinary work. The error due to cooling of the oil after leaving the outlet tube may be neglected at low temperatures, but requires correction at temperatures near 100° C Any observed variation in instrumental constants at different temperatures is probably due to the last mentioned error. Glycerin is not reliable for standardising viscometers, as it possesses some internal structure which is broken down by repeated passing through the instrument Samples of rape oil, on the other hand, have been found to vary 27% in viscosity. The highest velocity of flow for which the Redwood instrument can be used, or critical velocity, is 30·4 seconds Tables of factors are given for the comparison of Redwood, Saybolt and Engler viscosities. H. M.

THE OXIDATION OF PETROLEUM HYDROCARBONS.—W. V. Piotrowski and W. Jakubowicz. *Petroleum*, 1923, **19**, 5–9.

The authors review the different methods adopted by previous experimenters for the estimation of aromatic hydrocarbons in Polish petroleum. A Boryslav crude petroleum of 0·858 sp. gr., 16° C. cold test, 9·30% paraffin content, and 51–52° C. melting point was tested in Hess's apparatus. The fraction up to 80° C. contained 2% of aromatic compounds, and the fractions 80–90°, 3%; 90–100°, 4%; 100–110°, 6%; 110–120°, 8%; 120–130°, 10%; 130–140°, 12%; and 140–150°, 13%. The cracked fraction from 90–110° contained 18%, from 110–120°, 24%; from 120–130°, 30%; 130–140°, 34%; and 140–150°, 38% of aromatic compounds. H. M.

THE PRODUCTION OF PETROLEUM FOR 1921 IN RUSSIA.—*Russian Mining Journal*, p. 38, 1922.

Baku district	156,653,000	poods.
Grosny ,,	80,823,000	,,
Ural Caspian district	4,332,000	,,
Total	241,808,000	,,

Number of Wells in Exploitation—

Baku district	1053
Grosny ,,	155
Ural Caspian district	33

Amongst the producing wells of Baku 20 are spouting wells, 94 are pumped by compressors, and the rest are exploited by bailers.

In the Grosny district 9 spouters, the rest by bailers.

New borings for the year 1921.

Baku district	2861	sagens.
Grosny ,,	1043	,,
Ural Caspian district	94	,,

THE PRODUCTION OF PETROLEUM IN RUSSIA FOR JANUARY—MAY, 1922.—*Mining Journal*, 3–5, 1922 (Russian).

Baku district	60,871,000	poods.
Grosny „	28,888,000	„
Ural Caspian district	2,298,000	„
Total production	92,057,000	„

CRACKING.

CRACKING AND HYDROGENATING HYDROCARBONS.—G. F. Forwood and J. T. Taplay. E.P. 190,284 of Oct. 7, 1922.

Oil is atomised and passed with steam over heated charcoal or other carbonaceous matter. The carbon is heated to a temperature sufficiently high to decompose the steam but not to injure the oil. Any permanent gas produced is freed from sulphuretted hydrogen and carbon-dioxide and returned to the retort containing the carbon to hasten the passage of the oil through the apparatus. When high boiling oils are being treated, the formation of carbon in the retort is such as to necessitate periodic removal of the excess. This can be obviated if the process is alternately worked with high and low boiling oils, as in the latter case carbon is removed from the retort. T. A. S.

HYDROCARBON OILS: PROCESS AND APPARATUS FOR TREATING.—R. B. Day, Assr. to Day's Petroleum Process Co. U.S.P. 1,437,932-3 of Dec. 5, 1922; Application, Dec. 15, 1920.

The oil is heated to cracking temperature in tubes which are fitted with conveyor worms. The conveyors agitate the oil and also to propel through the tubes small metallic elements. The cracking tubes are set one above the other and arrangements are made for conveying the small metallic elements from the bottom of the system to the top when they have passed through the tubes. An expansion chamber is provided above the cracking tubes. T. A. S.

CRACKING APPARATUS.—E. C. Blasdell. U.S.P. 1,437,712 of Dec. 5, 1922; Application, April 7, 1922.

The apparatus consists of horizontal cylindrical still fitted with a column. Communication is provided between the top of the column and the top and middle of a second smaller column. Arrangements are also made for the return of liquid from the bottom of the second column to the still, and also to remove condensed liquid from the system. Valves are fitted in the pipes to control the pressures in the different parts of the system. T. A. S.

LIGHT HYDROCARBONS: METHOD OF OBTAINING FROM HEAVY HYDROCARBONS.—D. W. Hovey. E.P. 188,928; Application, Oct. 31, 1921.

The oil to be treated is atomised in a still which is practically filled with porous brickwork. The brickwork is heated by means of coils carrying superheated steam. A pressure of 25 lb. or more per sq. in. is maintained in the still and the residues may be withdrawn from time to time or continuously. T. A. S.

HYDROCARBONS FROM CRUDE PETROLEUM, SHALE OIL, OR GILSONITE: PROCESS OF CRACKING. J. L. Murrie. U.S.P. 1,435,652 of Nov. 14, 1922; Application, May 12, 1920.

The hydrocarbon oil, along with permanent hydrocarbon gas, is introduced into the lower part of a molten metal bath. The resulting vapours are condensed and heavy hydrocarbons and carbon impurities are drawn off the surface of the molten metal. T. A. S.

PETROLEUM: PROCESS OF TREATING.—R. Cross, Assr. to The Gasoline Products Co. U.S.P. 1,437,229 of Nov. 28, 1922; Application, Jan. 11, 1918.

Oil is circulated under pressure through a cracking tube and a reacting chamber, where it is maintained at cracking temperature Permanent gases and cracked oil are continuously removed from the reacting chamber. T. A. S.

CRACKING PATENTS.

Some interesting facts on cracking patents are disclosed in a series of articles describing litigation on processes published in *National Petroleum News* **14**, No. 37–48.

The Standard Oil Company of New Jersey are seeking to prevent the Pure Oil Company from using the Cross Process as being an infringement on their Ellis Patent, and are themselves cited as defendants in an action brought by the Universal Oil Products Company, in respect of the Burton Process being an infringement on Dubbs' Patent.

In the former case Carleton Ellis filed his original claim for patent on October 4th, 1913, and on November 24th, 1920, divided his claims and filed the application on which the Patent in question, No. 1,415,232, was granted.

In this Ellis claims "a process of making gasoline substitute which comprises passing a kerosene or heavier oil through a heated zone, wherein the oil is brought to a decomposing temperature in a single passage, and passing the products at a slower rate through an enlarged passage-way without increasing their temperature and without permitting the temperature to drop below a decomposing temperature, and maintaining super-atmospheric pressure upon the oil in its passage through the heated zone and the enlarged passage-way."

In reply, the Pure Oil Company deny any infringement of the Ellis Patent, and state that the same process had been patented in the United States, Germany, Great Britain and Russia, prior to the application of Ellis, *viz.*:

United States Patents.

419,931.	Dewar and Redwood ..	January 21st, 1890.
342,564.	Benton	May 25th, 1886.
477,153.	Pielsticker	June 14th, 1892.
755,309.	Nikiforoff	March 22nd, 1904.
1,049,667.	W. M. Burton	January 7th, 1913.

German Patents.

166,723.	De Cleroq	January 10th, 1906.
85,884.	Nikiforoff	March 17th, 1896
221,898.	Reichmann	May 12th, 1910.
88,122.	Dvorkovitz	September 11th, 1896.

British Patents.

1706.	Pielsticker	1882.
1411.	Ragosin	1898.
26,910.	Wilton	1907.
11,079.	Laing	1890.
16,881.	Testelin	1908.
8336.	H. A. Kittle	1909.

Russian Patent.

175.	Schuchow and Gawrylow ..	1891.

Publications are quoted by Schuchow and Garwylow, in *Journal Society Chemical Industry*, 1893, p. 590, and *Zeit. fur Ang. Chem.*, 1893, p. 230.

Defendants further state that the process has been known to, and used by, certain persons named prior to the application being granted, and in his application Ellis disclosed less than the whole truth relative to his said invention.

Much more important, however, is the latter case, in which the validity of the Burton Patents is challenged, and incidentally, the practical monopoly of the Standard Oil Co. to produce gasoline from heavier distillates.

Legal action by the Universal Oil Products Company against the Standard Oil Company was commenced in 1916, but owing to the War has only recently come up for hearing. Mr. Dubbs in his original application, dated March 20th, 1909, made only two claims for a dehydrating process for improvement in treating an emulsified oil. In accordance with action taken by the patent examiner the application was amended and broadened at various later dates, until the patent No. 1,123,502 was granted, dated January, 1915. This contains 11 claims, 7 of which deal entirely with dehydration and remaining 4 with a process for treating hydrocarbon oil under heat and autogenous pressure applied both to still and condensers. No mention is made of any claim to cracking heavy oil into gasoline. The Dewar and Redwood Patent appears to have been the chief difficulty and Dubbs, in his amendments, got over that by emphasising the fact that pressure is not maintained by pumping extraneous gas into the still. Although used as a dehydrating process on a small scale since 1908, cracking was not attempted on the Dubbs' process until 1916, and since then only to a comparatively small extent.

Dr Burton filed his application in July, 1912, and the basic patent, No. 1,049,067, was granted in January, 1913. In this is claimed "the method of treating liquid portions of the paraffin series of petroleum distillation having a boiling point of upwards of 500° F., to obtain therefrom low-boiling point products of the same series, which consists in distilling at a temperature of from about 650° to about 850° F., the volatile constituents of the said liquid, conducting off and condensing the said constituents, and maintaining a pressure of from about 4 to 5 atmospheres on the said liquid of the said vapours throughout their course to, and while undergoing condensation." As in Dubbs' claim so in this, the Dewar and Redwood patent was cited as showing prior invention, but Burton showed that whereas he relied on still gases to maintain the required pressure Dewar and Redwood used extraneous gases, and in so doing raised the pressure to such an extent that the

product contained an excessive proportion of olefinic bodies. Burton further claimed that the effect of pressure on the condenser as well as the still avoided the formation of compounds of the ethylene series with subsequent loss on refining. This claim has been shown by Brooks, Bacon, Padgett and Humphrey, *Journal Ind. Eng. Chem.*, March, 1915, to be a misstatement of fact, that it is immaterial where the valve is placed, and that olefines are produced under the conditions prescribed by Burton.

Notwithstanding Burton's original claim, the Standard Oil Co.'s counsel admits in his opening statement that the Burton process as used to-day is substantially the Dewar and Redwood process with added improvements not having the slightest relation to where the valve is or how far the pressure goes. This he states was never a point of novelty, and the process never depended on that.

These suits are only examples of many others in contemplation and it is not surprising that public opinion is strongly in favour of drastic action such as annulment by the Attorney-General of all existing cracking patents. The status of the process of cracking heavy oils into gasoline is in the worst muddle imaginable, and new claims only add to the confusion. Litigation will not clear it up and answer the question as to what, if anything, is patentable in the fundamental application of heat and pressure to the manufacture of gasoline.

The growth of the independent Refiner, his ability in the future to make gasoline to meet the public's demand, depends entirely on his freedom to instal a cracking process. He cannot do this until he knows who holds rights, if any, and what these rights are. S. B.

Lubricating Oil and Fuel.

Motor Fuel: Process of Manufacture.—L. de Florez, Assr. to Motor Petr. Co. U.S.P. 1,437,045 of Nov. 28, 1922; Application, Dec. 4, 1917.

Hydrocarbon oil, not containing fractions sufficiently volatile for use as motor fuel, is cracked by heating it under high pressure. The portions having a higher boiling point than about 200° C. are removed by lowering the pressure on the gases. The lighter vapours are condensed at atmospheric pressure and any gases uncondensed are subjected to a pressure of about 10 atmospheres and absorbed in the light distillate under that pressure. T. A. S.

Liquid Fuel: Process of Manufacture.—G. E. Gray. U.S.P. 1,438,823 of Nov. 12, 1922; Application, July 2, 1920.

Kerosene, or light hydrocarbons heavier than kerosene, are mixed with acetone. The mixture is sprayed over pieces of inert material to increase the liquid surface and brought into contact with a gaseous hydrocarbon. T. A. S.

Lubricants: Improvements in.—Technical Research Works, Ltd. and R. G. Pelly. E.P. 188,364 of July 7, 1921.

The lubricating properties of oils are improved by the addition of polyhydric alcohols or phenols in which the hydroxyl groups have been partially replaced by fatty or other acid radicles. The emulsifying

properties of the lubricating oils are increased. The addition of 5 per cent. of the mono- or di-glyceride of oleic acid to a mineral lubricating oil reduces its coefficient of friction. T. A. S.

LIQUID FUEL: PROCESS OF MANUFACTURE FROM PEAT.—F. N. Nicholls and D. Brown. E.P. 188,469 of Sept. 22, 1921.

Liquid fuel suitable for use in high speed internal combustion engines is manufactured from peat by a fermentation process. A layer of peat 12 in. to 18 in. thick is moistened with water and allowed to decompose for 3 to 12 days according to atmospheric conditions. Growth is then stopped by heating and drying the peat which is subsequently ground and extracted with hot water (3½ gals. of water to 50 lb. peat). The fibrous residue is discarded and the aqueous extract is boiled for 3 to 4 hours. After cooling to 40° F. yeast or other fermenting agent is added and fermentation allowed to proceed at 90°—98° F. The scum is then removed and the liquid distilled at about 220° F. and under a pressure of two atmospheres. Double distillation yields a product suitable for use in internal combustion engines either alone or mixed with colya or rape oil distillate. The fermentation product boils at about 170° F., has a flash point of about 90° F. and a specific gravity of about ·825. T. A. S.

FUEL FOR INTERNAL COMBUSTION ENGINES.—B. H. Morgan. E.P. 189,715 of Dec. 7, 1922; Application, April 22, 1922.

The formation of acid from the combustion of alcohol-ether mixtures in internal combustion engines is overcome by the addition to the fuel of 0·2 to 5 per cent. of castor oil. The miscibility of the fuel with petrol or similar fuels is increased. T. A. S.

VISCOUS OILY COMPOSITIONS.—Plauson's (Parent Co.). E.P. 186,950 of 1921.

Wax or crude bitumen, such as ozokerite, beeswax, montan, carnauba or Japan wax is treated with an aldehyde such as formaldehyde or furfural in presence of an alkali with or without the addition of a ketone such as acetone or methyl-ethyl-ketone, or a divalent or trivalent alcohol such as glycerol or glycol. The product, after removal of the alkali by washing, may be added to mineral tar, animal or vegetable oil, yielding viscous or paste-like products which may be used as lubricants or cosmetics; or emulsified with water, forming salves or leather-dressing compositions. The product may be mixed with 20-30% of paraffin wax, ceresin, ozokerite, or the like without affecting its properties. Instead of aldehydes, the polymerisation products of aldehydes or substances such as hexamethylene-teramine may be used. T. A. S.

MOTOR FUEL.—Riedel Akt.-Ges., Berlin. E.P. 184,785 of Oct. 11, 1922; Conv., Aug. 15, 1921.

A fuel for internal combustion engines consists of a hydronaphthalene, an aliphatic hydrocarbon of low boiling point, and methylated spirit blended with a small proportion of ether or alcohol of high boiling point. A suitable fuel consists of tetrahydronaphthalene 30 parts petrol or light petroleum 35 parts, methylated spirit 30 parts, and amyl alcohol 5 parts. T. A. S.

VACUUM DISTILLATION OF OIL.—W. L. Gomory. *Pet. Age* **10**, No. 7, 117-20 (1922).

The vacuum, steam and dry distillation methods for the preparation of lubricating oils are compared. Vacuum distillation produces a lubricating stock of higher viscosity, fire and flash tests, negligible C content and lighter colour in higher yield than is obtained with ordinary steam distillation methods. The expense of installing vacuum plant is easily offset by the economies and superior products obtained. T. A. S.

DISTILLATION AND REFINING.

SEPARATION OF HYDROCARBONS VAPOURS BY PARTIAL CONDENSATION.—H. C. Smart, assr. to Standard Oil Co. U.S.P. 1,437,689 of Dec. 5, 1922; Application, March 25, 1919.

The hydrocarbon vapours are passed through a fractionating column and are made to take the form of a thin annular stream on the walls of the column. The outer wall of the column is cooled by a stream of air, flowing in a counter current direction to the hydrocarbon vapours. The column is divided into sections, and the condensed liquid is removed from each section without allowing it to flow through the lower sections. The cooling effect is regulated by controlling the air stream. T. A. S.

SOME RECENT DEVELOPMENTS IN THE MANUFACTURE OF NATURAL GAS GASOLINE.—F. E. Hosmer and F. M. Seibert. *J. Ind. Eng. Chem.*, Vol. 14, No. 12, 1106–11.

Recent developments in the recovery of gasoline from natural gas have been very rapid. In 1904 two plants only were operating, recovering 400 barrels per annum; in 1920 650 plants recovered 483 million gals. of gasoline. Natural gas is classified as wet or dry according as it comes from gas or oil wells. Natural gas is varied in composition, a typical wet gas containing methane 38·8%; ethane, 32·6%; propane, 21·1%; butane-pentane, 5·8%; hexane, 3·7%; and a typical dry gas, containing methane, 87·1%; ethane, 5·0%; propane, 6·5%; nitrogen, 1·4%. Of the wet gas 9·5% is recoverable as gasoline, 1000 cub. ft. yielding 95 cub. ft. of condensable vapour, equal to 3 gals. of gasoline. The gasoline is recovered by compression or adsorption processes, the latter being considered the most practical. Low-pressure compression systems, working at 70 to 125 lb., where additional cooling of the gas is brought about by evaporating gasoline under slight vacuum, the evaporated gasoline being added to new gas and removed by water cooling under low pressure, are more economical to work than the high-pressure expander systems working at 250 lb. Adsorption systems are of two kinds, using solid and liquid absorbents respectively. The absorbents used are activated charcoal, silica gel, crude oil or even low-grade gasoline. Plants using solid absorbents are claimed to be more economical in fuel. When crude oil is used as an absorbent it is necessary to cool it after leaving the absorbers, owing to the heat developed during absorption. Crude oil tends to absorb a certain amount of dry gas, but this can be overcome by suitable temperature control of the oil just before it leaves the absorbers. T. A. S.

TREATMENT OF PETROLEUM AND OTHER LIQUID HYDROCARBONS.—A. E. Dunstan and F. G. P. Remfry. E.P. 190,553 of Sept. 28, 1921.

Oils are desulphurised and decolorised by treatment with floridin or fuller's-earth, and then with bauxite alumina or other colloidal gel.

A kerosene with an initial colour of 10 Lovibond requires treating with half-a-pound of floridin before desulphurising with bauxite. A kerosene distillate which, without the preliminary floridin treatment, would require 1 lb. of bauxite per gallon to bring about effective desulphurisation only required ¼ lb. of bauxite per gallon after floridin treatment. The floridin is roasted before use at a temperature of 250–300° C., and used hot. T. A. S.

MANUFACTURE OF MATERIALS FOR ADSORBING CONDENSIBLE GASES AND VAPOURS.—B. Lambert. E.P. 188,786 of Aug. 18, 1921.

Iron hydroxide gel is formed by adding slowly with constant stirring a solution of 16% by weight of caustic soda to a solution of ferric chloride, containing about 16% by weight iron in the cold. The gel is allowed to settle out and is then washed with hard spring or river water, containing small amounts of dissolved matter, until free from soluble matter. Sodium or potassium carbonate, equal in concentration to the caustic soda, may be employed in place of the latter, whereby a gel having marked catalytic properties is obtained. Concentrated caustic soda does not give so good an absorbent gel as the dilute solution. The washed gel is pressed into slabs or sticks and dried slowly at 26–36° C., then broken up and dehydrated at 110–160° C., or up to 180° in vacuo. Condensible gases are readily absorbed, whilst fixed gases pass through the material. The former may be recovered by heating to their boiling point or by passing steam at a temperature above 100° C. F. G. P. R.

LIGNITE—THE DISTILLATION OF.—S. Ruhemann and O. Zeller. *Z. angew. Chem.*, 1922, **35**, 725–727

To study the heat changes in the distillation and coking of lignite an electrically-heated retort was employed, in which 500 g. of lignite could be uniformly heated by inner and outer heat elements, and the energy consumed measured Experiments were made with Meuselwitz lignite (braunkohl), of 5575 gross cal. value, 3·5% soluble in benzene, and with 10% of ash On slowly distilling lignite most of the distillate goes over up to 400° C, and the solid and liquid distillates are almost entirely driven off when 500° C is reached. In the test made 500 g. of lignite was heated to 500° C. and the amount of heat required to raise it to this temperature noted. The distillates were then returned to the retort, and the same heating process carried out. A comparison of the results in the two cases gave indications as to the degree of the exothermic or endothermic nature of the process. In similar experiments the temperature reached in 5 hours' heating was, in the first place, 520° C., and in the second place, 498° C., showing that the first operation, of distillation, was exothermic. At ordinary pressure it was found that solid paraffins came over at 250° C, while the first drops of oil, from water-containing lignite, came over at 150° C. The sublimed paraffin wax, after repeated crystallisation, had a composition of 84·95% carbon and 13·11% hydrogen, had an approximate m.p. of 65° C., and crystallised in scales from solution in hot glacial acetic acid or boiling alcohol. Examinations of the gases evolved were made in an apparatus in which 50 g. of lignite were heated by an air bath in a glass bulb. Free nitrogen was not evolved : at 200° C. hydrogen sulphide, carbon dioxide and small quantities of hydrocarbons were set free. Hydrogen was not given off up to 425° C., the highest temperature of the experiment. H. M.

TREATMENT OF OIL-FIELD EMULSIONS.—D. B. Dow. *Chem. and Met. Eng.*, 1922, **27**, No. 23. 1120.

Most oil companies employ chemicals for breaking water-oil emulsions in the field, but the centrifugal process is in rather general use in a number of fields. Laboratory tests showed that less emulsion was formed, and was easier to break when a large excess of water was present. With 10% of oil and 90% of water about 98% of the water separated out on standing, but with decreasing amounts of water an increasingly poorer separation took place. This is in accordance with observation on the fields. F. G. P. R.

OIL; METHOD OF TREATING.—E. M. Johansen, assr. to the Atlantic Refining Co. U.S.P. 1,438,764 of Dec. 12, 1922; application May 9, 1919.

The oil, either crude or reduced crude, from topping plants, is washed with soap solution. The washed oil is treated with acid and subsequently with alkali. T. A. S.

GASOLINE FROM NATURAL GAS; APPARATUS FOR RECOVERY.—J. B. Garner and S. C. Preston, arrs. to Hope Construction and Refining Co. U.S.P. 1,437,101–2 of Nov. 28, 1922; application A of Nov. 21, 1919, and B Feb. 14, 1920.

The gas is passed through an eddy in a stream of vapour absorbing liquid, and subsequently again through the liquid. T. A. S.

LIQUID HYDROCARBONS; PROCESS OF OXIDISING.—E. Zerner. U.S.P. 1,436,136 of Nov. 21, 1922; application Jan. 1, 1922.

The unsaturated bodies present in hydrocarbon oil are extracted by means of liquid SO_2. The extract, after removal of the SO_2, is oxidised by blowing through it oxygen containing gas. T. A. S.

ASPHALTIC PRODUCTS; PROCESS OF PRODUCING.—L. Kirschbraun. U.S.P. 1,436,450 of Nov. 21, 1922; Application Feb. 16, 1918.

Oil is distilled in a plurality of stills, superheated steam being injected. The vapours are passed through a succession of condensers. The primary condensers are kept at a temperature above the boiling point of water by passing through them the heated cooling water which has passed through the secondary condensers, the latter being maintained at a temperature below the boiling point of water. T. A. S.

REFINING ELEMENT AND METHOD OF MAKING SAME.—Z. Olsson, assr. of six-tenths to A. Rust-Oppenheim. U.S.P. 1,435,972 of Nov. 21, 1922; Application Sept. 15, 1920.

The refining element is made of bauxite and bentonite, and made up in the form of a spherical filter. In making the element a mixture of calcined bauxite (75%), bentonite (5%), and starch (20%), is heated to a temperature of at least 700° C. T. A. S.

HYDROCARBON STILL.—J. C. Pool, assr. of one-half to W. B. Poole. U.S.P 1,436,526 of Nov. 21, 1922; Application Feb. 18, 1920.

The still, which is fitted with fire tubes, is steeply inclined over a furnace setting. A settling tank, communicating with the still, is placed

outside the setting. The hot furnace gases pass upwards through the fire tubes, and then downwards, outside the still, to the stack.

T. A. S.

IMPROVED APPARATUS FOR DISTILLING COAL TAR, PETROLEUM AND OTHER LIQUIDS.—F. J. E. China and Burt, Boulton & Haywood, Ltd. E.P. 188,575 of Dec. 28, 1921.

Continuous stills in series are connected in such a manner that the generation of pressure in any one of them will not cause the contents of the still to be discharged. The stills are connected near the surface of the liquid by a horizontal pipe to one end of which is attached an open-ended T-pipe, reaching from the surface of the liquid nearly to the bottom of the still. The normal path of the liquid from one still to the next is through the lower end of the T-pipe, but any developement of pressure is released through the upper end of the T with minimum disturbance of the still contents. T. A. S.

IMPROVEMENTS IN THE FRACTIONATION OF CRUDE NAPHTHA AND APPARATUS THEREFOR.—G. E. Walker. E.P. 188,402 of Aug. 11, 1921.

Crude naphtha is heated by means of steam to a temperature of 200–300° C., the light and heavy benzol distillates being collected separately. The distillates are washed with acid and soda and then carried to continuous acting fractionating columns, where the commercial benzol, toluol and solvent naphtha are separated. These columns, as described in E.P. 121,748, are heated by steam and hot residues, which are passed through pipes in the lower parts of the columns. T. A. S.

BITUMEN; IMPROVEMENTS RELATING TO MANUFACTURE AND TREATMENT OF.—H. Burnice, from Bituminal Matita Roumania. E.P. 188,354 of July 4, 1921.

Bitumen is manufactured from bituminous hydrocarbons, such as crude oil, by distilling the crude oil to a temperature of 274° C., a small quantity of calcium carbonate having been previously added to neutralise the sulphuric acid formed during distillation to the residue sulphur is added, and the mixture is heated to 250° C. It is very important that during the primary distillation the temperature of the oil should not rise above 274° C. The quantity of sulphur necessary is determined by a small scale experiment, the addition of sulphur being continued until a bitumen of the desired consistency is obtained. The addition of too much sulphur adversely affects the ductility of the bitumen. The residues from certain Roumanian crude oil requires 7·4% sulphur.

T. A. S.

THE DESULPHURISATION OF CRUDE OIL DISTILLATES.—H. I. Waterman and D. W. Sissingh. *Chem. Weekblad*, 1922, **19**, 489–492.

When crude distillates are treated with sulphuric acid and washed with sodium hydroxide solution the sulphur content is considerably decreased. Experiments with five fractions showed a reduction of about two-thirds, as, from 0·74% to 0·27%, and from 3·31% to 1·16%. Refining with sodium plumbite solution and flowers of sulphur sometimes leaves the sulphur content unaltered, or even increases it. On redistilling such a refined fraction considerable quantities of hydrogen sulphide are given off, and the sulphur content reduced, as, from 1·3% to 0·42%. The "doctor

test," which consists in shaking with a solution of sodium plumbite and flowers of sulphur, the presence of sulphur being shown by the sulphur turning gray or black, is not applicable to Mexican crude distillates, which may give a negative test, though the sulphur content be as much as 1%. H. M.

RECOVERY OF GASES OR VAPOURS FROM ABSORBENT SUBSTANCES.—E. R. Sutcliffe and H. S. Raper. E.P. 188,723 of July 16, 1921.

Gases or vapours that have been absorbed by charcoal or other absorbent materials are recovered without suffering decomposition by passing steam, superheated to about 300° C. through the material. Some condensation of water takes place in the material, and in order to dry the latter the treatment with superheated steam is continued until the temperature of the issuing steam reaches 110° C. The material is then cooled by substituting saturated steam at 100° C. for the superheated steam and continuing this treatment until the temperature of the issuing steam has fallen to a few degrees above 100° C. By this means the condensed moisture does not rise above 3–4%, and the material is ready for re-use. F. G. P. R.

FRACTIONATION OF PETROLEUM; APPARATUS FOR.—H. P. Straus. E.P. 189,239 of Aug. 29, 1921.

The vapours from a still are carried to an insulated fractionating column, which consists of a number of superimposed chambers separated by perforated partitions, and partially filled with broken stone or metal balls. Pipes, fitted with traps are supplied to carry down to the chamber immediately below any condensate that cannot escape through the perforations. Each chamber is fitted with a vapour line, from which vapours may be drawn when desired. T. A. S.

OIL AND SHALE.

RETORT; FOR OIL SHALE.—W. Alenius. U.S.P. 1,438,421 of Dec. 12, 1922; Application May 20, 1921.

An externally-heated cylindrical retort is fitted with a longitudinal rotary agitator and conveyor. The conveyor consists of a shaft with spaced cross arms keyed on to it. L-shaped angle irons are wound spirally along the shaft, and fit into the outer ends of the cross arms. Perpendicular stops are fixed at intervals along the angle irons. The diameter of the conveyor is the same as that of the interior of the retort. Means are provided for collecting the gases arising from the agitator material. T. A. S.

PROCESS AND APPARATUS FOR THE EXTRACTION OF HYDROCARBONS FROM WOOD, SHALE, OR THE LIKE.—Plauson's, Ltd. E.P. 188,686 of May 17, 1921.

In order to avoid decomposition and loss due to overheating the material to be extracted is heated by means of direct application of a non-oxidising gas under a reduced pressure of 60–70 mm. of mercury

The material is fed through a vertical or horizontal tubular retort by means of a screw conveyor, gas-tight, rotating valves at each end enabling the material to be supplied and extracted without allowing air to enter the retort. Steam at 250–450° C., preferably admixed with 0·1–1·0% of hydrogen or purified water gas, is passed through the retort in counter-current to the material and thence through wire gauze filters to a receiver, vacuum pump, and condensers. The retort is covered with insulating material, and is not heated externally. The vacuum pump acts in two stages, compressing the extracted gases to just above atmospheric pressure, in order to facilitate condensation. By means pure colourless resins can be extracted from wood and clear, mobile, sulphur free oil from shale. F. G. P. R.

BITUMINOUS SHALE; IMPROVEMENTS IN FURNACES FOR UTILISATION OF.—S. V. Bergh and K. E. Larson. E.P. 188,693; Application May 20, 1921

The spent shale from continuous retorts is delivered on to a continuous chain stoker and passed under a steam boiler, where the heat of combustion of the residual carbon is utilised. The ash is delivered into a vertical pit and preheats the air used in combustion which is drawn through the hot ash by means of a steam ejector. T. A. S.

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Chlorinating Paraffin Hydrocarbons.—G. T. Koch, Assignor to Ohio Fuel Supply Co. U.S. Pat. 1,432,761. *Chem. Abstr.* **17**, 289. *Chem. and Met. Eng.* **27**, 1233.

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USE OF NAPHTHENIC ACIDS IN SOAP MAKING.—E. Shuck. *Mat. grasses* **14**, 6249. *Chem. Abstr.* **17**, 220.

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PREPARATION OF PETROLEUM FROM ROSIN.—S. Kawai. *Kógyô-Kwagaku Zasshi* (*J. Chem. Ind., Japan*) **25**, 1421. *J. Soc. Chem. Ind.* **42**, 257A.

LES COMBUSTIBLES LIQUIDS ET LEURS APPLICATIONS.—Syndicat d'Applications Industrielles des Combustibles Liquids. Gauthier-Villars et Cie., Paris.

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DISTILLING MINERAL OILS FRACTIONALLY WHILE STILL IN WELLS.—W. J. Knox. U.S. Pat. 1,433,956. *Chem. Abstr.* **17**, 467.

Abstracts.

Compiled by P. Dvorkwitz, Ph.D., H. Moore, M.Sc., A.I.C., F. G. P. Remfry, B.A., D.Sc., A.I.C., and T. A. Smith, B.Sc., F.I.C.

General.

Iodine Value of Petroleum Oils.—S. Kawai. *J. Chem. Ind. Japan*, 1922, **25**, 406–19.

The Wijs, Hubl and Hanus methods for the determination of iodine values were tried under various conditions on two samples of Akita oil and one of Nishiwara having sp. gr. of 0·9242, 0·9397 and 0·9162. By increasing the amount of solvent (CCl_4) an increase of iodine values was found. When the amount of oil is varied the iodine value changes reciprocally with the oil, but by varying the amount of iodine solution the values change proportionally with the amount. With all components kept constant the values increase with the temperature at which the reaction is carried out. Absorption increases rapidly during the first hour, after two hours is comparatively slow and the values continue increasing indefinitely with the time of absorption. The general nature of the curve of increase is the same for all the three oils used. The Hubl value was less than half that of Wijs, and Hanus slightly greater than the latter. All methods gave the same kinds of variations with varying conditions, and therefore the latter must be specified when giving the iodine value for petroleum oils. F. G. P. R.

Expansion of Oils: Means of Calculating.—G. H. McNair. *Petr. Age*, 1922, **10**, No. 5, 80.

A table of the coefficients of expansion of petroleum oils having gravities between 26° and 76° Be (sp. gr. ·897–·680) is given. By means of the formula V=Vt [C (T, observed. —T, standard)+1] where Vt=volume at the observed temperature T, and C=the coefficient of expansion, volumes can be reduced to any standard temperature.
F. G. P. R.

Determination of Specific Gravity of Petroleum Products.—E. Kroch. *Mat. grasses*, 1922, **14**, 6257–8.

To obtain d_{15} from d_t the following formula should be used:—$d_{15}=d_t[1+a\ (t-15)]$ instead of the Engler-Hofger formula of $d_{15}=d_t+a\ (t-15)$, which gives an error of 1–3 units in the third place of decimals.
F. G. P. R.

Results of Holding Back Pressure on Oil Wells.—T. E. Swigart. *Nat. Petr. News*, 1923, **15**, No. 3, 65–71.

Experiments were carried out, from September, 1921, to March, 1922, on two wells in Osage county, Oklahoma, to determine the effect on oil production of increasing the pressure within the wells by means of limiting the amount of gas escaping. The efficiency of the tests was determined by the ratio of cubic feet of gas escaping to each barrel

of oil produced as compared with this ratio when the wells were producing normally. The results of the experiments showed that in general a slight decrease in barrels per day was brought about by increasing the back pressure, but at the same time the gas production was largely diminished. The total amount of oil that could ultimately be obtained from a well would in all probability be increased by this procedure since the life of a well depends on the internal gas pressure in the sands. The proximity of other wells, however, limits the application of high back pressures owing to the oil being driven towards wells where lower or atmospheric pressures are employed. F. G. P. R.

THE PETROLEUM INDUSTRY OF TRINIDAD.—*American Government Report* by Consul H. T. Baker to U.S. Dept. of Commerce.

Twenty companies are operating, but most have only a small production or are not producing at all at present. Only three companies —Trinidad Leaseholds, Trinidad Central and British West Indies Petroleum Syndicate, have refineries and supply the entire local marke in gasoline, as well as doing export. The gasoline is of only medium quality, and the kerosene even worse as it contains a high percentage of asphalt. The total storage capacity in the island is roughly 1,820,000 barrels, of which Leasehold possesses 500,000 and United British Oilfields 440,000. The principal imports for 1921 were illuminating oils 113,790 gallons, lubricating oils 130,808 gallons, gasoline 752 gallons and greases 122,399 lbs. Production during the same period was 82,395,595 gallons, against 72,905,947 gallons in 1920 and 64,436,632 gallons in 1919. Exports of crude petroleum in 1921 amounted to 4,020,702 gallons, fuel oil 33,781,450 gallons, refined petroleum 1,993,821 gallons and motor spirit 2,032,977 gallons. F. G. P. R.

THE OXIDATION OF HYDROCARBONS, WITH SPECIAL REFERENCE TO THE FORMATION OF FORMALDEHYDE.—T. S. Wheeler and E. W. Blair. *J. Soc. Chem. Ind.*, XLII., 10, T. 81.

It has been shown (Bone and Wheeler, *J. C. S.*, 1902, 535; 1903, 1074) that between 400° and 500° C., methane and oxygen, in contact with a surface, react to form formaldehyde as well as carbon monoxide and carbon dioxide. In one experiment formic acid was also detected. The reaction is supposed to proceed through methyl alcohol, dihydroxymethane, formaldehyde and further through formic acid and carbonic acid, the latter substance breaking down to carbon dioxide and water. Dihydroxymethane is unknown and methyl alcohol has not up to the present been detected among the oxidation products of methane. Bone and Wheeler recovered formaldehyde up to 22% of the methane burned. The present authors have examined the best conditions for the production of formaldehyde from methane. The following considerations are of importance :—

1. The thermal decomposition of methane is slower than its oxidation rate; below 850° C. no complications arise from this source during periods of heating of 1 second.

2. The rate of oxidation is slower than that of ethylene. A temperature of at least 600° C. is necessary to obtain a reasonable amount of reaction in a short period.

3. The explosive limits of methane lie between 5 and 60% of methane; the ignition temperature of methane is 550° to 700° C.

4. To isolate formaldehyde the time of reaction must be short as it commences to decompose at 310° C. Curves are given showing the relationship between methane oxidised and temperature and also for

the decomposition of formaldehyde. Slow oxidation and a short heating period are the main factors in obtaining a good yield of formaldehyde as the decomposition of the formaldehyde is rapid at high temperatures. T. A. S.

REACTION BETWEEN ETHYLENIC HYDROCARBONS AND GRIGAND REAGENT.—H. Gilman and H. M. Crawford. *J. Am. Chem. Soc.*, 1923, **45**, 554–8.

Several workers have assumed that addition takes place at the ethylenic linkage between R.Mg. X compounds and ethylenic hydrocarbons. Gilman (*J. Am. Chem. Soc.* **42**, 1010) has proved that this does not occur in the case of ketones, and later stated that there was no known proved case of such addition taking place. A variety of hydrocarbons possessing ethylenic linkages were therefore tried, including ethylene, amylene, diallyl cyclohexane, cyclohexadiene, phellandrene, 1, 1-diphenyl-ethylene,-propylene,-amylene, 1, 4-diphenyl-butadiene, and tetraphenyl-ethylene. The method employed was to reflux the reagent (C_2H_5–Mg I) with a slight excess of the hydrocarbon and then estimate the volume of the ethane given off from a known quantity of the solution of the reagent. If the amount before and after refluxing was the same obviously no reaction had taken place. It was only in the case of phellandrene, 1, 1-diphenyl-ethylene,-propylene and -butylene that any addition appeared to have occurred, but on investigation this proved to be due entirely to traces of benzophenone, except in the case of phellandrene, where the impurity was not traced but practically all the phellandrene was recovered unchanged. It is therefore believed that no addition takes place with ethylene linkages. F. G. P. R.

CURIOUS CAUSES OF GASOLINE FIRES.—A. H. Gill. *J. Ind. Eng. Chem.*, 1923, **15**, No. 2, 140.

An account is given of fires and explosions that have taken place under curious conditions when using light petroleum distillates for various purposes, and an attempt is made to give reasons for their occurrence. Hair shampoo mixtures containing petrol, alcohol or ether have been the cause of severe burns, ignition having been started by the victim generating sufficient electricity by walking over a carpet to cause a spark between the hair and some near object. Similarly a motorist wearing a fur coat and rubber boots generated sufficient electricity by friction against the fur to generate a spark when approaching the automobile with nearly fatal results to himself and total destruction of the car. Silk garments that have been dry cleaned and not properly freed from petrol have also caused fires by sparking when being removed from the basket. Frictional electricity generated when pumping petrol has also caused fires, and there should be good metallic contact between the nozzle and, for example, the motor car when filling from a stand pump. F. G. P. R.

CONSTITUENTS OF A BORNEO PETROLEUM FRACTION BOILING 37°–81° C.—G. Chavanne. *Bull. Soc. Chim. Belg.*, 1922, **31**, 331–64.

All possible isomerides of hexane, and all hydrocarbons derived from cyclopentane and cyclohexane boiling within the above range, were found to be present. All the cyclic compounds, except ethyl-cyclobutane, are derived from five or six membered rings. The quantities of hexanes present are in the following order :—normal –, iso –, γ-methyl-pentane, $\beta\beta$-dimethyl-butane, $\beta\gamma$-dimethyl-butane. Of the saturated cyclic hydrocarbons cyclohexane and methylcyclopentane are the most

abundant. The critical temperature of solution in aniline, and the density, are stated to be important factors in studying complex mixtures of hydrocarbons. F. G. P. R.

OXIDATION OF PETROLEUM : ADDITION TO KNOWLEDGE OF.—W. V. Piotrowski and W. Jakubowicz. *Pet.* **19**, 5–9, 1-1.

Benzine fractions boiling from 60–150° C. give, when pyrogenetically decomposed, practically only unsaturated hydrocarbons. Aromatic hydrocarbons are only present in very small quantities. These are probably formed from the unsaturated hydrocarbons, this theory being borne out by the fact that the quantity and character of the latter are considerably influenced by the presence of catalysts. T. A. S.

FIRE RISK OF BENZINE.—A. Brun. *Schweiz. Apoth. Zeit.* **60**, 556, 12-10, 1922.

A fire risk is always present when dry benzol, petroleum ether or ether are poured from vessel to vessel or filtered. This is particularly marked when pouring from one glass vessel to another. T. A. S.

LABORATORY EXAMINATION OF PARAFFIN AND VASELINE.—E. Pyhala. *Pet. Z.* **18**, 1164–5, 1922.

The specific gravity of paraffin is determined by floating it in a mixture of alcohol and water and determining the specific gravity of the mixture. In determining the dropping point of vaseline with the Ubeholde apparatus it was found that variations in results could be avoided by first melting the vaseline on a glass plate and allowing it to congeal thereon. This makes it more homogeneous. T. A. S.

THE ABSORPTION OF ETHYLENE BY SULPHURIC ACID. THE PRODUCTION OF ETHYL ALCOHOL, DIETHYL SULPHATE AND LIQUID HYDROCARBONS.—A. Damieus. *Comptes rend.*, 1922, **175**, 585–8.

The preparation of ethyl alcohol from ethylene by absorption in sulphuric acid has already been shown possible on the industrial scale. The rate of absorption is directly proportional to the pressure and surface of contact. Agitation is very important. Addition of 2% of H_2O reduces the rate of absorption. Raising the temperature favours absorption, but above 60° C. a partial decomposition of the organic compounds formed takes place. The rate of absorption can be considerably increased by the addition of catalysts. The catalyst giving the highest rate of absorption was Cu_2SO_4 or CuCl. By varying the conditions with these catalysts, three different products were obtained—alcohol, diethyl sulphate and an oil having the properties of petroleum: With 1 to 5 parts of Cu_2SO_4 per 100 in the acid ethyl hydrogen sulphate was formed at low temperature giving on hydrolysis an almost quantitative yield of alcohol. By carrying out the absorption in 100% H_2SO_4 in the cold in the presence of H_g or $H_{g_2}SO_4$ the rate was greatly increased and an oily mixture of hydrocarbons separated of sp. gr. 0·77 with fractions boiling as follows :—110–200° C., 40% ; 200–215° C., 20% ; 215–300° C., 40%. T. A. S.

THE ADSORPTION OF PARAFFIN WAX.—H. Burstin. *Petroleum*, 1923, **19**, 119–122.

Boryslav crude oil of sp. gr. 0·858 and paraffin wax content by Holde 9·0% (solidifying point of paraffin 50° C.) was shaken for an hour with Frankonit and filtered. The filtered oil contained 6·7% of paraffin wax of 54° C. solidifying point. The powder and filter being extracted

with petroleum ether, the ether evaporated, the residue refined, and the wax precipitated by alcohol-ether, the latter was found to have a solidifying point of 36° C. The softer paraffins were adsorbed by the powder. A further experiment, in which the Frankonit was suspended in the oil in a bag, gave results: wax in untreated oil 11·0%, wax in treated oil 8·0%. On continuing the treatment for five days, the wax in treated oil was only reduced to 7·8%. Treatment at 100° C. did not lead to a greater adsorption of wax. When the amount of Frankonit was increased from 10% to 33% of the oil, no appreciable increase in adsorption was noticed, nor was the content of resinous substances reduced. A second treatment increased the amount of soft wax extracted from 30% to 39%. Treatment for a night with Kieselguhr reduced the wax content from 11·0% to 7·4%. Treatment with powdered fire-brick gave a similar result to treatment with Frankonit, and the extracted wax was almost entirely recoverable by boiling the powder out with petroleum-ether. Treatment with Frankonit under 30 mm. mercury vacuum reduced the wax content from 10·0% to 7·5%. In all these experiments the solidifying point of the treated crude oil was found to have risen, contrary to the observations of Rakusin. When, however, a gas oil from which all but the softer wax had been extracted was treated the solidifying point fell. Much less wax is extracted from distillates than from crude oil. H. M.

PETROLEUM SOAPS AND NAPHTHENE ACIDS.—E. Mashkileyson. *P. S. J.*, 1921, N. 1–4, 62.

The Baku and Grozny petroleum contains special organic fat "naphthene" acids, or naphthene soaps, which are mainly obtained by the action of an alkali upon the residue left after the distillation of kerosene. When there were plenty of fats for saponification purposes the alkali waters were emptied in Baku into the sea, and not very long ago some companies began to produce therefrom a product called petroleum soap (in Russian mylonapht). It has the aspect of a dark or yellow grease. This product is mixed with ordinary soap, and it possesses remarkable washing qualities, which were also noted in London. The price of a pood (40 lb.) of this substitute, which before the war was as low as two roubles, rose in 1918 to about 35 times this amount.

The Baku district can give about 670,000 poods (10,000 tons) of such a soap yearly, and the conjoint production of all the Russian oil districts must be about 1,000,000 of poods. Considering the actual shortage of animal and vegetable fats, all soapworks will soon be forced to use petroleum soap.

The author gives a short outline of naphthene acids and describes the results obtained by some investigators during the production of acids from oil. The pure naphthene acids are a colourless viscous liquid with a specific gravity reaching 0·98. The acids give ethers, dense glycerides, liquid chlorides and crystalline amids. There is in the literature no data about the absorption of iodine by naphthene acids. The author explains how to recognise the presence of these acids in the soap.

The properties of the acids obtained from the alkali waters left after the refinement of lubricating oils (Russian, Galician or Roumanian, were investigated too, and it must be said that the results obtained by Schwarz and Marcusson possess too high molecular and high iodine numbers. After having explained the fractional distillation of naphthene acids and described the industrial manufacturing of petroleum soap and naphthene acids, the author gives several analyses of different

substitutes and shows how naphthene acids are produced from petroleum soaps.

Professor Lydoff determined the percentage of naphthene acids and phenols in Russian crude oils. In Baku the residue was 1·12%; in Grozny, 1·24%; in Balachany Crude, 1·12%; in Grozny Crude, 1·30%; Bibi-Eibat, 0·83%. The specific gravity of naphthene acids are from 0·9567–0·9629 and represent $C_nH_{2n-2}O_2$. P. D.

CHLORINATION OF METHANE.—A. Schleede and C. Luckow. *Ber.*, 1922, **55**, 3710–26.

Chlorine and methane passed through electrically heated quartz tubes burns when molar proportions are employed, but the flame gradually dies out unless an excess of chlorine is used. With excess of methane no ignition occurs, and addition of chlorine is only quantitative if the temperature is kept above a certain point depending on composition and rate of flow of the mixture. The presence of catalysts, or an increase of 100° C. above this temperature, do not greatly affect the result. Hydrogen chloride is removed from the products of reaction by warm concentrated potassium hydroxide and the gases then condensed at —110° C. Fractional distillation in a high vacuum by Stock's method separates methychloride, but technical distillation must be employed for the other constituents.

The following results were obtained :—

(*a*) When the velocity of the gases per unit of heated surface exceeds a certain value, and the heating surface is not largely increased, steady ignition obtains with increase of production of higher products at the expense of chloroform.

(*b*) If the rate of flow is below the ignition value not much of the higher products are formed, but the relative proportion of chlorinated methanes is not in agreement with a step-wise reaction. The production of carbon tetrachlorides and higher products is only observed if the heating surface is enormously increased, as by the use of activated charcoal. No appreciable difference is obtained by increasing the temperature 100° C. or using catalysts such as ferric chloride, molybdenum or antimony pentachloride.

(*c*) If the rate of flow is considerably below the ignition temperature chloroform, carbon tetrachloride or bodies of similar boiling point are formed at the expense of methyl and methylene chlorides. The dissociation of methane and such changes as $CH_3Cl+CH_4=C_2H_6+HCl$, $CH_2Cl_2+CH_4=CH_2Cl, CH_2+HCl$, $CH_3Cl+CH_3Cl=CH_2Cl, CH_2+HCl$, and $CH_2Cl_2+CH_3Cl=CH_2Cl, CH_2Cl$ are considered the most probable reactions taking place. The preparation of methyl chloride from methane and carbonyl chloride (Hocstetter A., 1916, i., 625) appears to be due to the great dilution of the chlorine. F. G. P. R.

DISTILLATION AND REFINING.

THE ABSORPTION OF GASOLINE FROM NATURAL GAS.—I. R. C. Cantelo. *Can. Chem. Met.*, 1922, **6**, 177–9 and 196–200.

Compression and refrigeration methods are only profitable with gas when in intimate contact with oil, and containing more than 1 gal. of gasoline per 1000 cu. ft. Natural gas when in large quantities can be treated successfully by the absorption process when only one quart of gasoline per 1000 cu. ft. is present. Absorption is carried out at 15° F., higher temperatures giving lower yields, but increasing the gas

pressure improves the yield. Pentane, hexane, and heptane are the principal products extracted. Details are given of the method of calculating the amount of oil necessary to completely remove gasoline from gas. At 0° mineral seal oil will dissolve 1·45 times as much hexane as pentane and 1·28 times at 10°. Theoretically, gasoline can be completely removed at any temperature by washing with that amount of oil that will give a solution having a vapour pressure of pentane equivalent to the partial pressure of pentane in the gas. Increasing the pressure increases this partial pressure, and thereby proportionally decreases the amount of oil necessary for absorption. F. G. P. R.

Cost of Producing Natural-Gas Gasoline.—E. G. Sievers. *Chem. Met. Eng.*, 1923, **28**, 7, 297–8.

From the original experimental scale in 1900 in the U.S.A., production has grown to 450 million gallons in 1921. It was not, however, till 1910 that the industry assumed commercial importance, and in 1911 the output was 7½ million gallons. Wet gas is usually stripped in absorption plants, whilst dry gas, containing less than 1 gallon of recoverable gasoline per 1000 cu. ft., is put through compression plants, which, at present, are responsible for 75% of the total annual output. A combination of the two processes is now coming into favour. Small plants producing less than 1000 gallons of gasoline a day are now hardly profitable, and in most cases it has been found more profitable to consolidate small plants, and change over to the absorptive process. By attention to details and careful working the average yield was raised 0·2 gallons per 1000 cu. ft. from 1920 to 1921. An absorption plant treating 20 million cu. ft. per day, and producing 4000 gallons of gasoline is estimated to cost 110,900 dols.; a 1000 gallons production, 40,000 dols.; and a 10,000 gallons production, 450,000 to 500,000 dols. The working pressure is an important item, and a plant producing 4000 gallons at 10 lbs. pressure would cost 175,000 to 200,000 dols., whereas when employing the charcoal method, which can be operated regardless of pressure, for the same production the cost is reduced to 90,000 dols., and an absorption plant, 110,000 dols. The latter works out at about 6½ cents per gallon, and is about the minimum for operating absorption plants in the Eastern fields. F. G. P. R.

The Limit of Absorptive Power of Floridin.—M. A. Rakusin. *Petr. Zeit.*, 1922, **18**, 797–8.

One per cent. of petroleum oil free from paraffin wax, from Grosny, dissolved in a colourless light distillate, was decolorised completely by standing at ordinary temperature for 24 hours with 5% of floridin. The carbonisation constant was thereby raised from 18·75 to 100%, but the solution was not polarised. Larger amounts of floridin gave no greater effect. The effect of time on absorption was very marked. With duration of absorption of 24, 48, 72 and 96 hours 1% of floridin gave decolorisation of 25, 50, 75 and 100%; 2% floridin, 50, 75 and 100%; 3% floridin, 75 and 100%; 5% and 8% floridin, 100%. The original solution was the colour of weak coffee. F. G. P. R.

Contact Process for Filtering Petroleum Products.—H. L. Kaufman. *Nat. Petr. News.* 1923, **15**, 3, 81–2.

Clays found in California which, after an acid treatment, are suitable for the purification of petroleum are hallaysite, pyrophillite and mont morrillonite. The so-called Death Valley clay is of the first type, but

the last is said to have twice the efficiency of the other two. To prepare the clay it is ground with water to 200 mesh, digested with weak acid, dried and roughly ground to 100 mesh. The oil to be treated is first acid washed, and without any subsequent neutralisation with alkali is agitated with 1 to 20% by weight of clay, at a temperature of 250–350° F. The presence of water in the clay is desirable since this promotes agitation by the discharge of steam, which also prevents oxidation of the surface of the oil by forming a protecting blanket over it. One hour and a half is all that is required for raising the temperature of the oil, agitation, and sending the treated oil to filter presses. The used clay is approximately 50% efficient and can be mixed with fresh material for re-use before being sent for revivification by burning. Lubricating oils generally show a slight increase in viscosity after treatment, and are said to keep their colour better at high temperatures than those treated by acid and alkali. Emulsion difficulties when neutralising with alkali are also eliminated.

F. G. P. R.

THE RESOLUTION OF PETROLEUM EMULSIONS.—H. V. Dodd. *Chem. Met. Eng.*, 1923, **28**, 6, 249–53.

Two antagonistic forces are found at the interface between the phases of an emulsion—interfacial tension causing coalescence, and coherence of the film of emulsifying agent tending to resist coalescence. The stability of an emulsion depends on the relative magnitude of these opposing forces. There are therefore two methods of breaking an emulsion: (1) by attacking the emulsifying agent, and (2) by increasing the surface tension of the interface. In petroleum emulsions asphalt is probably the agent, the coherence of the film of which varies somewhat with the nature of the oil, being promoted by excess of light fractions. The dispersed water globules carry negative charges which decrease the surface tension at the interface, and when neutralised this tension is increased to such an extent that the asphalt films are no longer able to prevent coalescence, and the emulsion breaks. By the addition of electrolytes having strongly adsorbed positive ions, emulsions may thus be broken, provided that it is possible to convey the electrolyte through the oil envelope to the dispersed water. This can only be done when excessive amounts are present, and then only with difficulty, except when a small amount of some substance soluble in both phases is present to act as vehicle for the electrolyte through the oil envelope to the water. Acid with phenol has been found the most efficacious. Kerosene acid-sludge contains mutually soluble substances, but in insufficient quantity. If, however, phenol is added the mixture is more effective than phenol and pure acid. A certain amount of heat must generally be applied as well when employing this method. Only a very small amount of acid is necessary, but it is not yet known whether enough remains in the oil to cause injury to pipe-lines and plant.

F. G P. R.

CLARIFYING OILS AND SOLVENTS.—N. E. Wills. E.P. 189,701 of 1922.

The oils are agitated with a mixture of water, glycerin, and a chloride or hydrochloric acid. Suitable proportions for benzine are a mixture of 5% glycerine, 5% of calcium chloride or hydrochloric acid, and 90% of water, and for lubricating oils, 10% glycerin, 10% chloride and 80% water. The grease and dirt remain with the clarifying agent and may be run off. When dirty the agent may be filtered through sand, etc., and re-used.

F. G. P. R

ROTARY DISTILLING DRUMS.—Maschinen Fabrik Augsburg-Nurnberg Aktg. E.P. 174,069 of Jan. 10, 1922 (Convention, Jan. 17, 1921.)

In rotary distilling drums for the distillation of oil-shale, coal and the like, heat is usually applied externally and steam admitted through the axle of the drum, whereby it merely passes over the surface of the charge. In order to pass it directly through the mass of material the steam is admitted to rib-shaped turning bars mounted on the internal wall of the drum. These bars are connected through valves to a header, which can rotate in the steam main, and trip-bars are so arranged as to open the valves only when the steam pipes are buried by the charge in the drums. F. G. P. R.

PURIFICATION OF MINERAL OILS.—C. R. Ehlers. E.P. 184,991 of July 29, 1921.

Resinous, tarry and bituminous substances are removed from mineral oils by agitation with 30–70% of an aromatic amine, such as toluidine, xylidine, aniline, etc., having a benzene nucleus. After separating off the amine solvent the refined oil may be treated with steam to remove any traces of amines. Anthracite coal-tar distillates may be used, or these may be nitrated and then reduced to form amines. F. G. P. R.

OILS AND TARS—APPARATUS FOR CHEMICALLY TREATING.—A. B. Foster. E.P. 190,754 of Aug. 23, 1921.

A closed vessel, having a cone-shaped bottom, is provided towards the upper part with nozzles fixed at right angles to one another, so that oil and chemical reagent, being forced separately through the nozzles, impinge on and atomise one another. A baffle plate is also fixed opposite the mouths of the nozzles, against which the atomised stream of mixed oil and reagent impinges and is further mixed. By connecting two vessels in series the partially spent reagent from one may be mixed in this way with crude oil entering the second, whilst the partially-treated oil from the latter is similarly treated with fresh reagent in the former vessel. F. G. P. R.

CONDENSERS—IMPROVEMENTS IN.—Heenan and Froude, Ltd., and G. H. Walker. E.P. 191,175 of Oct. 11, 1921.

In condensing apparatus, in which vapours pass from a vapour chamber through water-cooled pipes to a second vapour chamber, there is the risk of water leaking into the chambers through defective joints of the pipes. In order to prevent this a second wall is supplied to enclose the cooling water, thus leaving an air space between the water and the vapour chamber by means of which any water leaking through the second wall can be withdrawn. F. G. P. R.

IMPROVED PETROLEUM PREHEATER AND FRACTIONATING STILL.—Stirling Boiler Co., and W. W. Watt. E.P. 191,465 of Oct. 12, 1921.

Petroleum is caused to pass in one direction through a system of tubes, which are heated by the hot gases from the boiler or other furnaces travelling in the opposite direction. The tubes extend between a system of upper and lower vessels, and are situated in a flue through which the heating medium passes. Each upper vessel is fitted with a scrubbing tower to return heavy fractions to the stills. Superheated steam is admitted into both upper and lower vessels through perforated pipes. Baffle plates are situated between the sets of tubes to ensure a proper distribution of the heating medium, and that it impinges against the tubes in a counter-current direction. T. A. S.

ABSORBING GASES—APPARATUS FOR.—Farbwerk vorm. Meister, Lucius, and Bruning. E.P. 187,223.

For the removal of condensable liquids from gases, such as benzol from illuminating gas, the gas is passed through activated charcoal contained in a ring-shaped sieve drum which is divided radially by a series of imperforate partitions, and can be rotated. The whole is contained in a closed vessel provided with suitable openings for entrance and exit of gases and steam. The rich gas is made to pass inwards, through the charcoal to the centre space, and after being thus stripped, passes outwards through the opposite side of the sieve. By rotating the drum-sieve the charcoal ladened with liquid comes opposite steam pipes in the centre space, by means of which the charcoal is freed from the absorbed liquid, and passes on to be dried by the stripped gas passing out from the centre space. The operation is thus continuous without the need of removing the charcoal for revivification. F. G. P. R.

TAR DISTILLATION—IMPROVEMENTS IN.—S. W. G. Wikner and Newcastle-upon-Tyne and Gateshead Gas Co. E.P. 191,295 of Jan. 27, 1922.

Tar is dehydrated in a vessel, in which it is kept at constant level, and is heated by a steam coil placed just below the surface of the tar. The vapours formed are removed through a coil which is immersed in the tar, the open end of which is above the surface of the tar. The tar thus acts as a condenser and heat is conserved. After dehydration the tar is passed through preheaters, where it is heated by the vapours from a battery of stills, and finally through a pitch-residue preheater. It is then passed to the first of the battery of stills. Using this method the output of the plant is doubled. T. A. S.

RECOVERY OF ALUMINIUM CHLORIDE FROM PETROLEUM RESIDUES.—A. McD. McAfee. Canadian P. 222,306–7–8–21 of Aug. 15, 1922.

(6) The sludgy residue is treated with a volatile solvent, such as alcohol, carbon tetrachloride, etc., and the solvent removed, leaving the aluminium chloride revivified.

(7) After extraction of oily matter by solvents the aluminium chloride is leached out with water, and heated to produce hydrochloric acid and aluminium oxide. The latter is heated with carbon and hydrochloric acid to reform the chloride.

(8) The residues are roasted with a limited amount of air to form aluminium oxide, which is then electrolysed to metallic aluminium.

(21) The residues are heated to 950–1800° F., and the released aluminium chloride condensed. Al_4C_3 may be added to the residue before heating, and chlorine may be passed through the remaining residue.

F. G. P. R.

RECOVERY OF NATURAL GAS GASOLINE.—E. A. Starke. U.S.P. 1,439,921 of Dec. 26, 1922 (Appl. Sept. 12, 1921).

The natural gas is brought into intimate contact with an absorbing oil by passing the gas and the oil both into the suction side of a centrifugal pump. The gasoline is recovered from the absorbent oil by distillation.

T. A. S.

PETROLEUM OIL—PROCESS FOR REFINING.—J. C. Black. U.S.P. 1,447,118 of Feb. 27, 1923 (appl. June 20, 1922).

Fractional condensation of the vapours obtained on distilling oil is brought about by bringing the vapours in contact with liquid hydrocarbon of approximately the same composition as the fraction it is desired to separate. T. A. S.

PETROLEUM—APPARATUS FOR DISTILLING CRUDE.—C. B. Forward. U.S.P. 1,442,935 of Jan. 23, 1923 (appl. June 13, 1917).

Hydrocarbon oils are forced by means of a pump through a long series of pipes contained within two inter-communication steam chambers connected with a source of superheated steam, travelling in counter current to the stream of oil. The preheated oil then passes, together with steam, into a vessel wherein heavy oils are deposited and withdrawn, whilst the lighter oils and steam pass over to a condenser. Means are provided for controlling the pressure within the vessel through the discharge connexions for the heavy residuum, and at the condenser end. F. G. P. R.

DEHYDRATOR—VARIABLE FIELD.—H. C. Eddy. Assr. to Petroleum Refining Co. of California. U.S.P. 1,442,608 of Jan. 16, 1923 (appl. June 25, 1921).

Two plate electrodes are suspended horizontally within a closed vessel, the lower one being attached centrally to the end of a vertical liquid-feed pipe, whilst the upper is suspended on a movable chain. The latter is connected through a lever action with an electro-magnetic device, whereby the electrodes are caused to separate whenever the current flowing between them exceeds a predetermined value. Means are provided for drawing off water and liquid from the bottom and top of the vessel. F. G. P. R.

CONTINUOUS DISTILLATION—METHOD OF AND APPARATUS FOR.—F. M. Hess. U.S.P. 1,443,742–3 of Jan. 30, 1923 (appl. May 8, 1919, and Feb. 17, 1921).

The liquid to be distilled is caused to flow by gravity down a zig-zag pipe or channel, composed of flat double-walled plates connected by curved plates which are also double-walled. Superheated steam is passed upwards through the double walls composing each side of the pipe. The whole system is enclosed within a vessel which is connected at the top with a condenser and at the bottom with a second similar vessel, and so on, the liquid flowing down from the top vessel to the lowest, whilst the steam ascends in counter current. Thus fractionation of progressively higher boiling liquids takes place in each descending vessel. The crude cold liquid is employed to condense the vapours by passing downwards from the uppermost to the lowest condenser, thereby being preheated before entering the top distilling vessel. The hot residue from the lowest distilling vessel is pumped through ascending heat exchangers, through which the unvapourised portions from each distilling vessel pass on their way to the next lower vessel. F. G. P. R.

DEHYDRATOR.—F. W. Harris. Assr. to Petroleum Rectifying Co. of Califor. nia. U.S.P. 1,440,828 of Jan. 2, 1923 (appl. Dec. 12, 1921; orig. appl. Sept. 18, 1919).

Oil emulsified under pressure, is made to pass through a tank in which an electric field is maintained. Means are provided for interrupting the field when the fluid pressure in the tank is reduced. T. A. S.

OIL—METHOD OF TREATING.—C. F. Kennedy. Assr. to The Atlantic Refining Co. U.S.P. 1,439,171 of Dec. 19, 1922 (appl. May 24, 1919).

Oil which contains wax is washed with soap solution before separating the wax. The concentration of the soap solution is such that emulsification is avoided, and as a result of the treatment the wax is more easily separated. T. A. S.

WAX—METHOD OF TREATING.—T. G. Delbridge. Assr. to The Atlantic Refining Co. U.S.P. 1,438,985 of Dec. 19, 1922 (appl. May 10, 1919).

Wax, before sweating, is washed with soap solution. By this means colloidal solids are removed and a wax obtained whose crystals are larger and more uniform. T. A. S.

EMULSIONS—METHOD OF PRODUCING.—E. E. Werner. U.S.P. 1,445,427 of Feb. 13, 1923 (appl. Feb. 11, 1922).

Liquids of different specific gravities are caused to emulsify by first being withdrawn from the bottom of a container, so as to establish a vortex therein, and thereby developng a flow of the upper and lower liquids together through the outlet. The stream of mixed liquids is then directed against the smooth face of a rapidly revolving disc, thereby dispersing the lighter within the heavier, and returned to the container in a tangential direction in order to develop a rotary motion therein.

F. G. P. R.

APPARATUS FOR DEHYDRATING AND CLEANING OILS.—R. G. Jones. Assr. to Brown Process Co. U.S.P. 1,440,832 of Jan. 2, 1923 (appl. Feb. 7, 1921).

A container partially filled with water is fitted with concentric internal cylinders arranged so that the annular space can be heated. The oil to be treated is passed upwards through the central cylinder and then made to pass downwards through the water. T. A. S.

DEHYDRATING PETROLEUM EMULSIONS BY OSMOSIS.—W. Meredith. Assr. to Petroleum Rectifying Co. U.S.P. 1,440,835 of Jan. 2, 1923 (appl. Feb 14, 1921).

Emulsion and water are separated by a porous diaphragm, and a direct current is passed from the emulsion to the water. T. A. S.

ELECTRICAL DEHYDRATOR.—W. C. Eddy. Assr. to Petroleum Rectifying Co. U.S.P. 1,440,774–6 of Jan. 2, 1923 (appl. A and B, Dec. 19, 1921; C, Dec. 27, 1921).

A. The dehydrator consists of a tank whose circular inner surface forms one terminal of an outer electric field. A stationary structure in the tank has a circular outer surface forming one terminal of an inner electric field. Another stationary structure is provided whose outer surface forms one terminal of the outer electric field, and the inner surface one terminal of the inner field.

B. The dehydrator consists of a tank containing one stationary live electrode, and a movable earthed electrode, which is turned by the flow of liquid through the dehydrator.

C. A tank is fitted with two electrodes. Means are provided for delivering emulsion below the upper electrode and water below the lower electrode. T. A. S.

CRACKING.

SUCCESS OF CALORISING IN OIL REFINING.—B. L. Jarrett. *Oil Gas J.*, 1922, **21**, No. 24, 82–4.

When steel tubes, etc., are calorised by being coated with aluminium, it is said to be possible to increase the length of runs in oil-cracking by 20%. The corrosive action of sulphur and its compounds, as well as of carbon on the steel is prevented. F. G. P. R.

CONVERTING HYDROCARBONS OIL INTO OILS OF LOWER BOILING POINT.—R. Cross. E.P. 191,318 of March 21, 1922.

Oil is passed continuously, under pressure, through a series of pipes heated in a furnace to such a degree that cracking temperature is attained at or about the time the oil leaves the furnace. It then passes into a lagged reaction chamber, of relatively large size compared to the tube furnace, wherein the same pressure is maintained and no substantial loss of heat takes place. By means of a gas release valve on the reaction chamber the pressure is maintained at such a height that only those liquids which have a critical temperature lower than that obtaining in the chamber can become vapours. Thus, very little beside permanent gases is found in the vapour stage, and cracking takes place substantially entirely in the liquid phase in the reaction chamber. A suitable temperature and pressure may be 750° F. and 750 lbs. per sq. in. The oil is withdrawn continuously from the reaction chamber in such quantities as to maintain a constant level of liquid therein. Passage through the entire system occupies about 20 minutes, and gas oil thus treated will furnish around 30% gasoline. F. G. P. R.

PRODUCTION OF LOW-BOILING HYDROCARBONS FROM TAR OILS.—M. Melamid. E.P. 171,367.

Tar oils, containing a large proportion of hydrocarbons, are heated with hydrogen under pressure, tin being used as a catalyst. The presence of the tin allows low pressures and temperatures to be used. Satisfactory results are obtained if the material is placed in a tin-lined pressure vessel, hydrogen admitted until a pressure of 10 to 20 atmospheres is obtained, and then heated for some hours at 250–320° C. By this means 80–90% of the oil is converted into material boiling below 200° C., and the residue is suitable for lubricating purposes. T. A. S.

HYDROCARBONS—PROCESS OF PRODUCING—OF LOW BOILING POINT.—M. Melamid. E.P. 171,390 of Nov. 9, 1921. (Int. Conv., Nov. 11, 1920; addn. to E.P. 171,367; Int. Conv., Nov. 11, 1920.)

In place of heating tar-oils in the presence of tin and hydrogen, mineral oils and hydrocarbon oils, other than tar oils, are subjected to the same process with the production of similar low-boiling products.
F. G. P. R.

ALUMINIUM CHLORIDE—PROCESS OF RECOVERING; USED IN CONVERSION OF HIGH-BOILING TO LOW-BOILING OILS.—E. C. R. Marks (communicated by Hoover Co., U.S.A.). E.P. 192,106 of Aug. 22, 1921.

Aluminium chloride used in converting high boiling to low boiling oils (E.P. 189,200), *J.S.C.I.*, is removed from the contact chamber when the proportion of asphaltic and carbonaceous matter in the salt-hydrocarbon compound amounts to 25%, and is passed by way of a preheater to a furnace, wherein the aluminium chloride is distilled off, together with volatile oils, and returned to the contact chamber. Hydrochloric acid or chlorine may be passed over aluminium metal contained in a part of the furnace, whereby any loss of chloride is made up. F. G. P. R.

HYDROCARBON OILS—PROCESS FOR CONVERTING HIGH BOILING—INTO LOW-BOILING POINT OILS.—E. C. R. Marks (communicated by the Hoover Co., U.S.A.). E.P. 189,200 of Aug. 22, 1921.

Oil, preheated to the required temperature, is sprayed over aluminium chloride maintained at the same temperature by circulating oil from the preheater around the reaction chamber, which is composed of a series of

vertical tubes communicating between two chambers. Means are provided for circulating the oil through the reaction chamber, for condensing the light products formed, and regenerating and returning to the reaction chamber the spent aluminium chloride, together with any make up necessary in a continuous system. F. G. P. R.

HYDROCARBON OILS, COAL TAR, AND THE LIKE—PROCESS FOR CRACKING.—Gulf Refining Co., W. F. Garagher, and W. A. Gruse. E.P 174,885–7–8 of Feb 15, 1923 (appl. Jan. 14, 1922; conv. Jan. 15, 1921).

The formation of carbon deposits in cracking stills is prevented by circulation of the oil. The circulation is brought about by an application of the air lift principle. The still is fitted with eduction tubes into the bottom of which a small quantity of gas, *e.g.*, steam, inert gas, or gases produced by the cracking, is delivered, thus causing a circulation of the oil. The apparatus may be fitted to vertical, horizontal or continuous stills. In 174,087 the eduction tubes are replaced by longitudinal baffles, between which the inert gas is delivered to produce circulation, and in 174,088 transverse baffles are used. The erosive effect of the oil on the still walls may be increased by the addition of a little sand, gravel or metal filings to the oil, thus keeping the walls free from carbon. A more regular heating of the oil is claimed, better yields of gasoline resulting T A. S.

LIGHT HYDROCARBONS FROM HEAVY HYDROCARBONS—PROCESS FOR THE PRODUCTION OF.—C. R. Burke. E.P. 190,751 of Aug. 22, 1921.

Cracking of heavy hydrocarbons is brought about by distillation under pressure, which is produced by means of the condensed distillate acting in the form of a release valve. A chamber of larger size than the still is attached to the upper part of the latter by means of a long narrow, conical neck. Vapours from the still condense in the upper chamber, and are partially prevented from returning to the still by the pressure of the vapour in the latter, and owing to the narrowness of the neck connecting the two. Light vapours escape through the liquid seal and pass to a condenser, whilst heavy condensate gradually returns to the still to be recracked. At the end of the distillation the residue in the still is of much lighter colour than the original charge, owing to deposition of carbon and colouring matter. F. G. P. R.

PETROLEUM AND OTHER HYDROCARBONS—APPARATUS FOR CRACKING.—C P. Dubbs. Assr. to Universal Oil Products Co. U.S.P. 1,440,772 of Jan. 2, 1923. (Orig. Appl. Nov. 27, 1916; this appl. June 23, 1917.)

Preheated oil is passed through a series of retorts heated to cracking temperature. The vapours formed are passed through dephlegmators, condensed oil being returned to the retorts. The apparatus is maintained at pressures higher than atmospheric. T. A. S.

HEAVY HYDROCARBONS—METHOD OF CRACKING.—U. S. Jenkins and S. Schwartz. U.S.P. 1,440,996 of Jan. 2, 1923. (Appl. Nov. 4, 1918.)

Oil is supplied to a series of pressure stills, pressure distillate being taken off from each still. The gravity of the oil in each still is kept constant. The oil taken from the last still is of so high a gravity that it does not give a profitable yield of gasoline. T. A. S.

OILS—APPARATUS FOR THE CONVERSION OF.—J. H. Adams. U.S.P. 1,445,281 of Feb. 13, 1923. (Appl. Sept. 30, 1915.)

Oils are cracked under pressure in the presence of superheated steam by passing the two together through a series of horizontal pipes enclosed in a furnace. Steam is introduced through preheated pipes lying within the oil pipes and above removable pans for collecting carbon deposits. The cracking tubes are in open communication with the condensers, condensation thus taking place under pressure. F. G. P. R.

HYDROGENATING CARBONACEOUS MATERIAL—METHOD AND APPARATUS FOR.—T. Ulke. U.S.P. 1,445,423 of Feb. 13, 1923. (Appl. Feb. 23, 1922.)

Fluid products are obtained from carbonaceous material such as coal, peat, lignite, oil shale and the like, by hydrogenating at a high temperature and pressure such as 400° C. and 100 atmospheres, in an autoclave by means of a solid hydrogen-yielding compound, which will have a residue of alkali-carbonate such as sodium formate. The resulting vapours are led through condensers, the uncondensed portion after being freed from ammonia being passed through red-hot carbon to reduce carbon dioxide to monoxide. The alkaline residue from the autoclave is leached with water to recover alkali-carbonate, which, after being cautisised, is treated with the above carbon monoxide and alkali formate thus regenerated. F. G. P. R.

HYDROCARBON OILS—APPARATUS FOR CONVERTING.—W. F. Muehl. U.S.P. 1,444,128–9 of Feb. 6, 1923. (Appl. Aug. 17, 1922.)

Oil of high-boiling point is forced through a vertical tube still at such a rate under such super-atmospheric pressure that it is raised to cracking temperature, but remains in the liquid phase. The oil then passes to a horizontal, lagged digester, wherein the temperature of the oil remains substantially unaltered; cracking takes place and carbon is deposited. Means are provided for scraping the carbon to one end of the digester, and removing it without agitating the oil. Light portions of the oil are here vaporised and pass to a dephlegmator, whilst the heavier portions are led to a second digester, also in communication with the dephlegmator. Fresh oil is used as a cooling medium in the dephlegmator, thence overflowing to the second digester and passing with unchanged oil back to the tube still. Super-atmospheric pressure is maintained on the oil and vapours throughout the process. F. G. P. R.

GASOLINE—PROCESS FOR PRODUCING.—E. M. Hyatt. U.S.P. 1,445,688 of Feb. 20, 1923. (Appl. Aug. 31, 1921.)

Fuel oil, kerosene, still residues, etc., are cracked after a previous treatment with chlorine. The oil to be treated is placed in a vessel capable of standing a pressure of 150 lb. per sq. in., and chlorine is then passed in to the extent of 1 to 10 lb. per 50 gals. of oil treated. The oil is then heated to 200–350° F. for 12 to 48 hours, a pressure of 75 to 150 lb. being generated. The HCl produced is now allowed to escape, and the temperature is raised to 650–750° F. The pressure rises to 75–150 lb. per sq. in., an escape valve allowing vapours to pass to a condenser. The valve is set so that the contents of the pressure vessel distil at the rate of 5–10 per cent. per hour. Cracking takes place in the vapour phase in the upper portion of the pressure vessel. The condensed vapours are passed to a still where the gasoline and kerosene are distilled off. The gasoline is acid and requires treatment with caustic soda. It is claimed that the process will produce 60–70 per cent. of gasoline from an oil of 60° Bé.
T. A. S.

CONVERSION OF AROMATIC RING COMPOUNDS INTO MOTOR SPIRIT.—A. S. Ramage. U.S.P. 1,439,976 of Dec. 26, 1922. (Appl. Jan. 28, 1921.)

Aromatic hydrocarbons are mixed with a paraffin or olefine hydrocarbon and passed with superheated steam over heated ferrous oxide or similar steam decomposing material. The operation is carried out at a temperature of 500–700° C., and the reactions involved are :—

1. The formation of nascent hydrogen from the steam by the oxidation of the ferrous oxide.
2. The dehydrogenation of the saturated hydrocarbons to unsaturated hydrocarbons by the ferric oxide formed.
3. The splitting up of the unsaturated hydrocarbons at temperatures of about 650° C. into simpler hydrocarbons such as ethylene.
4. The nascent hydrogen reduces the aromatic bodies to cycloparaffins. T. A. S.

HYDROCARBONS—APPARATUS FOR TREATING.—S. L. Gartland. U.S.P. 1,445,433 of Feb. 13, 1923. (Appl. Oct. 28, 1921.)

High-boiling hydrocarbon oils are vapourised at atmospheric pressure, the vapours withdrawn and compressed under conditions of temperature and pressure which can be regulated. The pressure is then suddenly released, whereby cracking takes place, owing to rapid expansion of the vapours. F. G. P. R.

PETROLEUM—PROCESS FOR TREATING.—L. C. Read. U.S.P. 1,445,040 of Feb. 13, 1923 (Appl. April 4, 1917.)

Hydrocarbon oils are heated to a temperature of approximately 2000° F., at a pressure less than atmospheric, whereby chemical conversion takes place. The vapours are then compressed, and the pressure subsequently released whereby further cracking is brought about. F. G. P. R.

MOTOR FUELS.

OXIDISED KEROSENE.—J. H. James. Carnegie Inst. of Tech. *Oil Engineering and Finance*, Vol. III., No. 61, 308.

Oxidised kerosenes cause less "knocking" on kerosene engines than straight kerosenes. The oxidised kerosene generates the same power as the ordinary kerosene, although the theoretical thermal value is one-eighth less. The higher efficiency is attributed to the more complete combustion obtained. The oxidised kerosene is obtained by the catalytic oxidation of low-grade petroleums. T. A. S.

TESTING FUELS FOR HIGH-COMPRESSION ENGINES.—S. M. Lee and S. W. Sparrow. *J. Soc. Aut. Eng.* **32**, 11–16 (1923).

For mixtures of benzol and gasoline in aviation engines the compression ratios and volume percentage of benzol are given as follows :—5 : 4, 0 ; 6 : 1, 30 ; 7 : 2, 65 ; 9 : 2, 85 ; 11 : 5, 100 ; 14 : 0, 100. Alcohol was found to be twice as effective as benzene at low compressions in preventing detonation. At high compressions where less gasoline was used in the fuel there appeared to be little difference between the effects of alcohol and benzene. Pre-ignition apparently depends on : (*a*) heat of compression ; (*b*) heat supplied by hot spots in the cylinders. The first is increased with increase in the compression ratio, the second is probably decreased. The change in heat of compression is calculable by the

formula $T_2 = T_1 r^{n-1}$, where n is 1.25—1.4, r is the compression ratio, T_1 is the absolute temperature when compression begins and T_2 the absolute temperature at the end of the compression. Engines may require different blends, owing to differences in design causing unequal tendencies to pre-ignition. T. A. S.

Sulphur Compounds in Motor Fuel.—J. C. Chatfield. *Nat. Pet. News.* Vol. XV., No. 11, 133.

Sulphur compounds in petrol, which give a positive doctor test, are stated to be uninjurious to automobile equipment. In refining by the litharge process, in which elemental sulphur is added to the petrol, a certain amount dissolves, and this free sulphur is detrimental to the copper and brass parts of automobiles. This free sulphur cannot be detected by the doctor test. The U.S. Government does not require that motor spirit shall pass the doctor test, and refiners generally consider it undesirable. T. A. S.

Gas from Petroleum (or other Hydrocarbons) and Water—Improved Apparatus for Producing.—E. and M. Robinson. E.P. 191,411 of Oct. 11, 1921.

Oil is pumped through a coil, which is surrounded by superheated steam. A similar coil in the same heating vessel is used for heating water. The oil is then run into the top of a vertical retort, into which the water is sprayed. The retort is heated by means of superheated steam. The vapours so produced are scrubbed and passed to a burner. T. A. S.

Alcohol—Improvements in Carburetting or Enriching.—J. M. A. Chevalier, P. Bourcet, and H. Regnault. E.P. 168,308 of Aug. 3, 1921. (Conv. Aug. 27, 1920.)

Alcohol for use in internal-combustion motors is increased in calorific power by the addition to 1000 gms. of alcohol of 200–500 gms. of resin spirit (mean sp. gr. 0.890) and 125 gms. of light acetone oil (b.p. 70–130° C.) to act as a "combiner." Where absolute alcohol is used it is not necessary to add acetone oil. F. G. P. R.

Vaporising Liquid Fuels—Improved Apparatus for.—P. Bachhofer and J. Müller. E.P. 171,700 of Feb. 19, 1923. (Convention Nov. 18, 1920.)

The fuel is vaporised by means of an improved nozzle, the tubular outer portion of which is cut to produce a thin annular lip. The lip is short and so thick that no deformation to produce a widening of the gap between the lip and the central core can take place under the pressure of the oil. When oil, vaporised from such a nozzle, is injected into air compressed to 12 or 15 atmospheres, ignition is spontaneous. This result is only achieved if the edge has the required rigidity and resistance to deformation. The apparatus may be used in conjunction with 2- or 4-stroke engines, either as the sole fuel source, or to enrich poorer mixtures. It may also be used for furnaces, or for the heating of incandescent mantles for illumination purposes. T. A. S.

Gases and Inflammable Liquids—Improvements in Storage of.—E. E. Petitpierre. E.P. 187,600 of Feb. 22, 1923. (Appl. Oct. 13, 1922; Conv. Oct. 20, 1921.)

Peat, particularly yellow peat, is a satisfactory material for packing containers used for compressed gases or highly inflammable liquids. The peat, either in its natural condition, or after suitable preparation,

by having been ground or compressed into lumps or reduced to peat wadding, is washed and dried and mixed with other fibrous or porous materials. It is then rammed or compressed by hand into the container. After filling the receptacle with peat, the material is saturated with acetone, or other inflammable solvent, in which acetylene is dissolved.

T. A. S.

Lubrication.

Testing Oiliness by Friction-Testing Machines.—W. H. Herschel, *Chem. and Met. Eng.*, 1923, **28**, No. 7, 302–3.

Oiliness has been defined as "the property which causes a difference in friction when two lubricating oils of the same viscosity at the temperature of the oil film are used under identical conditions." With complete film lubrication oiliness has little or no effect, but a change of bearing metal, high pressures, wide clearance, low speed or low viscosity tend to produce incomplete film lubrication where oiliness may be detected. It is fairly well agreed now that oiliness is due to some form of interaction between the lubricant and the bearing metal, and in the present paper it is considered that the bearing metals are constant, and that oiliness is a property of the lubricant. It is now generally recognised that a journal is eccentric in its bearings, and that eccentricity diminishes as speed increases; also, that friction varies with clearance and eccentricity which is a function of $\frac{\mu n d^2}{p \Delta^2}$ (*Sommerfield. Zeit. fur Math. und Physik*, 1904, **50**, 97); *i.e.*, a function of viscosity, speed and ratio of diameter to clearance, divided by the pressure. Hersey (*Trans. A.S.M.E.*, 1915, **37**, 182) is of the same opinion, provided the supply of lubricant is adequate. If Δ, the film thickness of a concentric journal, is replaced by 2Δ, the difference in diameter of bearing and journal, the above formula may be called Sommerfeld's criterion (S). With μ = viscosity in poises S will be dimensionless if the pressure p is expressed in degrees per sq. cm., the speed n in revolutions per sec. and Δ in any convenient unit. If the smoothness of the bearing remains constant the friction is a function of the viscosity, provided the pressure, speed and clearance are constant. With viscosity and speed both variable friction will be a function of μn, whilst with μ, n and p all variable, and clearance constant, friction will be a function of $\frac{\mu \eta}{p}$. It seemed preferable, however, to introduce the term $\left(\frac{d}{2\Delta}\right)^2$ and plot the coefficient of friction against S in order to emphasise the effect of clearance upon friction. The lowest coefficient of friction, f, at the transition point $= \frac{\pi}{2}\sqrt{2}\,\frac{(2\Delta)}{d}$ (1), occurs when S has a constant value of $\frac{\mu n}{p}\left(\frac{d}{2\Delta}\right)^2 = \frac{5}{24\pi^2} = 0{\cdot}0211$ (2). Hersey gives friction curves for ideal bearings, taking $\frac{2\Delta}{d}$ as ·001, ·002, ·004 and ·010, for which the values of f at the transition point are 0·00094, 0·00188, 0·00377 and 0·00943, and agree with the equation (1). His values of $\frac{\mu \eta}{p}$ vary as to the square of the clearance according to equation (2). Wilson and Barnard concluded that $\frac{\mu \eta}{p}$ at the point of minimum

friction varied with the oiliness. If this can be confirmed, or that S varies with the oiliness, journal friction machines might be used to measure oiliness. Clearance is generally unknown, but if known, probably changes by wear during the test. It is, however, desirable to introduce the term $\left(\frac{d}{2\Delta}\right)^2$ merely as a reminder that the results rest on an assumption regarding clearance. It is convenient to give $\frac{2\Delta}{d}$ the value of 0·001, but the true value should be used when known.

F. G. P. R.

Reclamation of Used Petroleum Lubricating Oils.—W. H. Herschel and A. H. Anderson. *No.* 223 *Tech. Paper, U.S. Bur. of Standards.*

The oil in the crank-case of an automobile engine usually becomes diluted with fuel residues, and corrosion in the crank-case is probably due to sulphur in the fuel. It may also be contaminated by products of cracking, and by carbon deposits which work their way past the piston rings. In other cases, as in the steam turbine, the oil increases in viscosity by use. Oil may be reclaimed by heating to 60°–71°, the addition of a coagulant, agitation and settling, or by blowing live steam into it, and treatment in centrifuge. A continuous by-pass filtering system, in which the oil is preliminarily heated, is employed for keeping turbine oil in good condition, the acidity developed in the oil tending to remain in the sludge. Motor oils may be reclaimed by blowing steam through them to drive off kerosene, adding a small quantity of soda, agitating and settling. The flash and fire points are higher for the reclaimed than the used oil, but not so high as for the new oil. The sediment in the reclaimed oil is much more than in the new oil, but the demulsification is good. A high acidity is found to be compatible with good demulsifying properties in the Californian oils, which are generally much higher in acidity than Eastern oils. Determination of the effect of variations in organic acidity of oils upon the inter-facial tension were made with Donnan's apparatus, in which drops of oil are released under water, and the number of drops counted. Blends of a class B oil with an Eastern oil of high acidity gave negative results in the corrosion test, and the demulsibility was zero for acidities of 0.33 mg. potassium hydroxide and over. Demulsibility was reduced to zero by the addition of 12·5% of old lard oil. A compound with 37·5% of old lard oil, with an acidity of 2·69 mg. potassium hydroxide showed corrosion. The addition of the lard oil showed a decrease in interfacial tension. In refining, the leaving of 0·1% of acid sludge in the oil reduces the demulsibility from 1200 to 300. The analysis of a turbine sludge showed that the ash contained 42·22% of silica, and it is difficult to believe that this came from the core sand, as the turbine had been in use for several years. Used oils should be judged by the same tests as new oils, and will be equally serviceable if they satisfy the tests. H. M.

Lubricating Oils from Lignite Tar.—J. Marcusson and F. Böttger. *Mitt. Materialprüf.*, 1922, **40**, 250–252.

Lubricating oils may be prepared from lignite tar either by condensing the light oils from the distillation process by chloride of zinc or by heating under pressure, or by working up low-temperature lignite tar with superheated steam. A condensation product had an open flash point of 198° C., against 171° C. for the low-temperature product. Both showed a rapid falling off in viscosity, with increase in temperature. The two oils had a high saponification value, 6 and 11·5, and a high

iodine value, 34 and 45, and the contents of sulphur were 2·7 and 1·4, respectively. The asphaltic substances separated by benzene were different from those obtained from petroleum oils. They were almost completely soluble in alcohol, were mostly saponifiable, and consisted of oxy-fatty acids. The phenols in the condensation product amounted to 1% only, in the low-temperature product to 4·2%. The acetyl value of the oils showed a considerable content of alcohols. The effect of hydrogenating lignite oils is to considerably increase their acetyl value, while with petroleum oils this value is only slightly increased. H. M.

THE INFLUENCE OF LOW TEMPERATURES ON MINERAL OILS IN TRANSFORMERS AND SWITCHES, AND COLD TESTS FOR THESE OILS.—G. Brühlmann. *Petroleum*, 1923, **19**, 69–72.

At low temperatures oils in transformers and switches, being semi-solid, present an obstacle to heat transference, and local overheating may occur, in spite of the low temperature of the mass. In transformers with oil circulation the pipes may present such resistance owing to the presence of oil of pasty consistency that the circulation may be seriously interfered with. This happens only when the transformers are exposed to the open air or are in unheated premises, and after being out of use for prolonged periods. With switches, this may lead to danger from fire or explosion, as the movement of the switch is retarded, and as the flow of the oil between the separated surfaces is slow. A switch filled with pasty oil was found to require 80% more time for putting out than was the case with fluid oil. The solidification point is defined as the temperature at which the oil ceases to obey the laws of fluids. An apparatus was designed for the determination of the solidification point. A pressure of 400 mm. of water acted upon the oil in a U-tube, and the time taken for the oil to rise 40 mm. was noted. On plotting results at varying temperatures the break in the character of the curve where the oil ceases to follow the laws of fluids gave an indication of the solidifying point. The viscosity constantly increased in a resin oil, and no solidification point could be fixed. In cases where the solidification point is not sharp, 1° C. is taken as 1 mm. on the abscissa, and 1 second time of flow is taken as 1 mm. on the ordinate, and the solidification point is taken at the point where the tangent of the curve forms an angle of 45° with the axis of the abscissa. H. M.

MANUFACTURE OF LUBRICATING PRODUCTS FROM WYOMING AND COLORADO CRUDE OILS.—L. C. Welch. *Proc. Colorado. Sci. Soc.*, 1922, **11**, 275–294.

A very complete description of the entire process employed in producing all varieties of lubricating oils and waxes from Wyoming and Colorado oils. The original paper should be consulted for details of plant and refining operations, in which there is nothing of outstanding novelty beyond the advocacy of high-speed centrifugal separators for the removal of wax. F. G. P. R.

DETERMINATION OF TAR NUMBERS OF TRANSFORMER AND TURBINE OILS.—F. Schwarz and J. Marcusson. *Petr. Zeit.*, 1922, **18**, 741–2.

The tar number is taken to be the amount of matter extractable by alcoholic caustic soda solution from the oils after being subjected to a period of heating. 50 gms. of oil are heated to 150° C. for 50 hours, cooled and again heated to 80° for 15 mins. under a reflux condenser with 50 c.c. of a solution of 50% by weight of alcohol, and 4% by weight of caustic soda. It is then shaken vigorously for 5 mins., allowed to

stand over night, and the caustic layer separated, filtered, measured and unchanged oil extracted by 30 c.c. of low-boiling gasoline. The gasoline extract is shaken with 10 c.c. of fresh alcohol-caustic soda solution, the latter added to the first alcohol caustic extract, and the whole then acidified with hydrochloric acid. The acid solution is extracted several times with benzol, which is then washed free from acid by water, distilled off, and the residue dried at 105° C. for 10 minutes. F. G. P. R.

USED LUBRICATING OIL—APPARATUS FOR RECLAIMING.—General Electric Co. Communicated to British Thomson-Houston Co. E.P. 191,547.

Oil, after being freed from dust and carbon, is allowed to drip over heating elements, which consist of electrical resistances in sealed bulbs. By the aid of gauze surrounding the resistances the oil is made to form a film and volatile portions are removed. Two heating elements, one above the other, are used, the upper one operating at 110–125° C. and the lower at 135–155° C. The apparatus is made up in a size and form suitable for garages. T. A. S.

SHALE AND PEAT.

NEW APPARATUS FOR EXPERIMENTAL DISTILLATION OF OIL-SHALE.—Wobling. *Braunkohle*, 1922, **21**, 453–4.

The retort consisted of an inclined porcelain tube heated in an electric oven, whereby larger yields of tar could be obtained than by the use of any other apparatus. 400 gms. could be distilled in 3–4 hours with the consumption of 2 kw.-hrs. The retort gases were freed from H_2S and CO_2 by $Pb(OAc)_2$ and KOH to simplify subsequent analysis. The percentage of light oils from most shales was small, whilst 30–40% boiled between 150°–250° C. Preliminary treatment with dilute H_3PO_4 prevented excessive resinification when washing with H_2SO_4. Heating the oils with zinc dust or distilling the shales with iron failed to reduce the sulphur content. F. G. P. R.

THE EXPERIMENTAL RETORTING OF OIL-SHALES.—M. J. Gavin. *Bull.* 210 *U.S. Bur. of Mines*, 1922.

The variation in the quality of oils produced in the distillation of shale with rising temperatures is not in the nature of fractional distillation. Rapid retorting is favourable for high yields of oil, slow retorting gives a smaller yield, but better quality. If shale is retorted rapidly a higher temperature is required to effect complete distillation. Rapid retorting gives a smaller amount of spent shale containing less fixed carbon, a larger amount of paraffin wax in the oil, more nitrogen in the oil and less in the spent shale. The effect of passing steam through the retort is to give an oil having the characteristics of oil produced at a more rapid rate without the use of scavenging media. In large retorts of the vertical type the quality and quantity of oil produced would be low without steaming. Reflux condensation is reduced to a minimum by steaming. With slow retorting the decomposition of bitumen to desirable products is more complete. In the small experimental retorts used, better oil is produced without the use of steam. When shale is broken into pieces the larger sizes yield more oil of a better quality than the smaller ones. The character of the products is materially altered by alteration in the design of the retort. The coking distillation of crude shale-oil increases the motor fuel fraction, with a decrease in its unsaturated content, and causes a loss in paraffin wax. The evolution of gas from shale begins at a

temperature below that of the first oil production, and continues after oil production has ceased. The first gas is rich in hydrogen sulphide. With some shales 20% of the gas is hydrogen sulphide.

H. M.

DISTILLATION OF CARBONACEOUS MATERIAL—APPARATUS FOR.—T. W. S. Hutchins. E.P. 190,819 of Oct. 1, 1921.

A tubular, rotatable iron retort, heated externally, is provided internally with an iron tumbler which may extend the length of the retort. The tumbler is in the form of a bar having four radial fins, and thus in cross-section forms a rectangular cross. As the retort rotates the tumbler is carried upwards until it overbalances and falls to the bottom. By means of the blow thus delivered caking of material on the sides of the retort is prevented. A gas-tight joint at the end of the retort is obtained by means of a corrugated flexible diaphragm attached to the retort, and kept in close contact with a collar on the exit pipe by means of springs. F. G. P. R.

LIQUID FUEL—IMPROVEMENTS RELATING TO.—J. A. Leadbeater. E.P. 191,201 of Oct. 20, 1921.

Five to ten parts of wet black peat, thirty parts of bituminous coal or other carbonaceous material, and one part of tallow or soap are ground together in a disintegrating machine to a fine or colloidal state. The ground material is then mixed with fifty-nine to sixty-four parts of crude oil and again ground in the disintegrating machine or passed through a Plausons colloidal mill. The resultant liquid fuel is suitable for combustion under boilers in the form of a spray either along or upon an ignited bed of coal or coke. T. A. S.

OIL-BEARING SHALE—METHOD OF TREATING.—H. R. Straight. U.S.P. 1,441,542 of Jan. 9, 1923. (Appl. July 31, 1918.)

After heating to remove the more volatile constituents, the shale is placed in a container and oxygen admitted to the central portion. The container is agitated to prevent the shale fusing and the combustion removes the remaining volatile portions of the shale. T. A. S.

SHALE—APPARATUS FOR COMBINED SOLVENT AND DESTRUCTIVE DISTILLATION TREATMENT OF.—D. T. Day. U.S.P. 1,447,296 and 7 of March 6, 1923. (Appl. (A) Dec. 7, 1920; (B) Aug. 30, 1922.)

(A) Shale is fed into a bath of oil and conveyed from thence to conveyor tubes, in which it is destructively distilled. The hydrocarbon oil produced is passed to the oil bath, from which vapours are removed.

In (B) the patent is extended to cover the similar treatment of oil containing earthy material. T. A. S.

PETROLEUM DIVISION

OF THE

AMERICAN CHEMICAL SOCIETY.

Abstracts of Papers submitted at the New Haven Meeting, April 2–7, 1923.

1. Determination of Volatility of Mineral Oil.—C. J. Rodman, Westinghouse Research Laboratory, E. Pittsburgh, Pa.

The factors influencing the volatility of an oil are: surface exposed, interchange of atmosphere above the oil, and temperature. The tendency of an oil to creep along the surface of the containing vessel varies with the kind of oil, thereby greatly altering the volatility. To get away from the non-uniformity in surface area the following method has been devised which works excellently for non-drying oils.

Procedure.—Filter paper of any good grade (corresponding to Whatman's No. 40) is rolled up upon itself until a paper rod is made which is about one centimeter in diameter and 10 centimeters long. A fine copper wire wound spirally about ten turns around the roll holds it in a compact form. A free end of the wire suffices for its suspension within a glass stoppered weighing bottle. The roll is dried first to constant weight at 100° C. Two cc. of oil are run on the filter paper roll so that the entire surface is wet by the oil. The roll is weighed, then suspended in the electrically controlled drying oven which is held at 100° C. The results are reported in loss of weight per centimeter squared surface area per hour. For a good transformer oil the loss should not exceed 0·003 gram centimeter squared per hour.

2. The Thermochemical Balance in the Cracking Re-action.—Jacque C. Morrell and Gustav Egloff, Universal Oil Products Co., Riverside, Ill.

The method of attacking the problem in the present research is based upon the laws of the conservation of matter and energy. Since the total quantity of matter is the same before and after the reaction, the difference in heat energy of the total starting material and the total final products when burned to carbon dioxide and water is a measure of the type of thermochemical reaction.

Examination of the data obtained shows that the cracking re-action for a Mexican crude distillate is neither exothermic or endothermic within the error of the experimental measurements. A Mid-Continent Topped Crude on the other hand showed that 1·64% of the total heat energy was given up during the re-action. A mixed Illinois-Kentucky fuel oil gave similar results to that obtained with the Mid-Continent Topped Crude.

The conclusion drawn from the above is that the cracking re-action may be slightly exothermic with some types of oils.

3. Coke Formation in the Cracking Process.—Jacque C. Morrell and Gustav Egloff, Universal Oil Products Co., Riverside, Ill.

The formation of coke as a function of type of oil and cracking conditions is shown, although it may be said that no fixed generalizations can be drawn from these data.

The densities of cokes from the cracking of various types of oils have been determined. The calorific values have also been obtained as well as other information concerning the chemical characteristics of the coke. Extraction experiments have been carried out with various solvents using a representative type of coke with the end in view of finding the maximum extractable matter in the cokes as taken from the stills.

4. THE COLOURING MATTER OF CRACKED GASOLINE.—B. T. Brooks and H. O. Parker, Mathieson Alkali Works, New York City.

5. NOTE ON THE DOCTOR RE-ACTION FOR SULFUR.—B. T. Brooks, Mathieson Alkali Works, New York City.

6. FERROUS SELENIDE AS A CONTACT CATALYST FOR CRACKING.—Thomas Midgley, Jr., and Carroll A. Hochwalt, General Motors Research Corporation, Dayton, Ohio.

It was found that selenium, which exerts a powerful influence on combustion, in the form of ferrous selenide is a contact catalyst for cracking petroleum oils. A very striking increase in the nitratable material and a corresponding decrease in the critical temperature of dissolution with aniline were obtained. The product obtained was yellow in colour. A slight increase in gasoline yield was obtained.

7. THE RELATIVE EFFICIENCY OF CATALYSTS IN THE PYROLYSIS OF PETROLEUM HYDROCARBONS.—Elmer H. Haux, Thomas Midgley, Jr., and James R. Withrow, General Motors Research Corporation and Ohio State University.

A survey was made of the effects of contact substances on the cracking of petroleum hydrocarbons in the vapour phase. Metallic oxides were found to have very little catalytic action whereas nickel and the sulfides of the iron group tend to decrease the yield of lighter fractions. Elementary selenium was found to have the most active catalytic "cracking" effect. The degree of cracking and the formation of aromatic compounds increase rapidly with a rise in temperature, in the presence of all contact substances. The temperature of critical dissolution with aniline was found to be a satisfactory indication of the degree of cracking.

8. HIGHER ALCOHOLS FROM PETROLEUM OLEFINES.—W. W. Clough and Carl O. Johns, Research Division, Development Department, Standard Oil Company of New Jersey.

This paper gives a general description of the method used in manufacturing higher alcohols from the unsaturated hydrocarbons found in refinery gases. The series of alcohols which is obtained includes 2-propanol, 2-butanol, 2-pentanol, 2-hexanol, 2-heptanol, and 2-octanol. The properties of the alcohols and some of their derivatives are given. The first three alcohols of this series are now manufactured and sold in commercial quantities.

9. THE INTERFACIAL TENSION BETWEEN PETROLEUM PRODUCTS AND WATER. E. M. Johansen, Atlantic Refining Company, Philadelphia, Pa.

Two different types of apparatus for the determination of the interfacial tension between petroleum products and water or aqueous solutions have been designed and are described as to construction and operation. Data are given, showing the degree of accuracy obtained in the determinations of tensions, the values of tensions for a number of products, and the factors that influence the value of the tensions. Suggestions are made as to the information that may be gained by aid of tension determinations and for what purposes such information may be used.

10. Both Temperatures of Viscosimeters of the Saybolt Type.—W. H. Herschel, Bureau of Standards, Washington, D.C.

Unless an efflux viscosimeter is practically all submerged in a bath of adequate size, there is doubt whether the actual temperature of test, or average temperature of liquid in the outlet tube during the timed interval, is equal to the nominal temperature of test or bath temperature. Where, as in instruments of the Saybolt type, the bath temperature is not equal to the nominal temperature of test, it becomes a difficult matter to determine suitable bath temperatures. This paper describes tests in which the actual temperature in the outlet tube was examined by thermocouple, in order to procure evidence in regard to proper bath temperatures.

11. Changes in the Viscosity of Mineral Oils on Standing.—Eugene C. Bingham and G. Raymond Hood, Lafayette College, Easton, Pa.

The question has been raised whether mineral oils maintain a constant fluidity suitable for use in calibrating viscometers. A sample of pale mineral oil, measured by W. L. Hyden in 1921, showed a fluidity of 0·9243.

We have redetermined this sample, using the same instrument, recalibrating and checking the constants given; and obtained values showing an increase in fluidity of 1·36%.

To discover if this increase could be accelerated, the oil was heated to 100° C. for two hours, cooled and remeasured. The average increase in fluidity was 0·40%.

Tests for the properties of a plastic solid apparently show that the discrepancy in fluidity cannot be attributed to this cause.

The authors conclude that it is inadvisable at present to employ mineral oils for calibration purposes.

12. Direct Determination of Dew-Points of Gasoline-Air Mixtures.—W. A. Gruse, Mellon Institute, Pittsburgh, Pa.

An apparatus has been developed for the direct determination of the dew-points of gasoline-air mixtures of the compositions employed in internal-combustion engines. Curves showing variation of dew-point with composition of mixture are given for several typical gasolines. The process is suggested as a direct means for the evaluation of motor fuels.

13. Thermal Decomposition of Shale-Oils under Pressure.—C. W. Botkin, New Mexico A. and M. College, State College, N.M.

Two shale-oils were heated at about 300° C. under the pressure of the gases produced by the decomposition (maximum 300-350 lb.). Eighty-two and 92% of oil was recovered, containing respectively 7 and 26% of oils distilling to 150° C.; an increase in this fraction of 100 and 380% respectively. The percentage of saturates in the light fraction from the cracked oils is about 66; an increase of approximately 25%. A third shale-oil was decomposed under conditions similar to the Burton process (laboratory apparatus, 500 cc. of oil), yielding 65·4% of oil by weight, 23·9% of which distilled below 150° C. and 56% below 210° C.; an increase of 29% in the last-named fraction. This fraction (to 210° C.) contained 56% of saturated compounds or 16% more than similar fraction of the original crude oil.

The date of these preliminary experiments indicate that by pressure decomposition the yield of crude gasoline from shale-oils may be increased to 45% and that this treatment much improves the refining qualities. However the yield and quality of the crude motor fuel fraction is not so high as obtained from the usual petroleum oils under the same conditions.

14. MOTOR FUEL SYMPOSIUM JOINTLY WITH GAS AND FUEL SECTION.—Van H. Manning, Chairman.

"Fuel Requirements of Internal Combustion Engines," S. W. Sparrow, National Bureau of Standards.

"Economic Aspects of Motor Fuel Supply from Petroleum," F. W. Lane and A. V. Bower, U.S. Bureau of Mines.

"Refinery Problems Affecting Motor Fuel Supplies," Frank A. Howard Standard Oil Co., N.Y.C.

"Suggestions on Absolute Alcohol as a Possible Constituent of Motor Fuels," H. A. Gardner, Institute of Industrial Research, Washington, D.C.

"Coal-Tar as a Source of Fuel for Internal Combustion Engines," Wilbert J. Huff, The Koppers Co., Pittsburgh, Pa.

PAPERS READ

AT THE

FIRST ALL-RUSSIAN PETROLEUM CONGRESS

(REFINERY SECTION)

(DECEMBER 31, 1921–JANUARY 10, 1922, MOSCOW).

(P.S.J. 1922. N. 5–6, p. 314.)

(Abstracted by P. Dvorkowitz, Ph.D.)

ACTIVITIES OF THE PARAFFINOID-FUEL COMMITTEE OF THE SUPREME TECHNICAL COMMITTEE OF THE PEOPLE'S COMMISARIAT OF WAYS AND COMMUNICATIONS. By Prof. A. V. Sapozhnikoff.

The Committee is engaged in evolving the most efficient methods of treating Grozny paraffinoid petroleum so as to render them suitable for use as locomotive fuel. With this object in view the Committee has been investigating the properties of paraffinoid-petroleum products, and the influence exerted on their physical properties by changes of temperature.

In the course of these investigations the Committee has, by a series of experiments, established "a method of determining the maximum freezing-point," tested the influence of admixtures of paraffin scale on the fluidity of petroleum products, tested the mixtures of paraffin petroleum products with various non-paraffin products, especially in regard to flash-point and freezing-point. The principal result of these investigations has been to establish the importance of preliminary treatment by heat, which determines the length of the period during which they can be preserved in a liquid and flowing state, and the rapidity with which paraffin scale may be separated therefrom when cooling.

The Committee has collected data on the question of the danger of fire and explosion (in cases when paraffinoid petroleum products have been diluted with lighter or more volatile oil fractions), also meteorological data relating to the temperatures characteristic of the principal regions where paraffinoid fuel is likely to be used.

METHOD OF DETERMINING FREEZING-POINT OF PETROLEUM PRODUCTS. By I. O. Luchinski.

The existing methods, in addition to giving contradictory results, offer this drawback, that if a maximum freezing-point has been determined by one of them this is by no means a guarantee that freezing will not occur at higher temperatures. This is due to the influence of the treatment by heat to which the petroleum product may have been subjected previously. This makes it necessary to evolve a standard method which would determine once and for all the maximum freezing-point for any given petroleum product.

The method suggested by the writer differs from Holde's method by the treatment of the petroleum product by heat, the chief object of which is to counteract the influence of superheating that may have been preserved in the product since the refining of the same. With this object

in view the petroleum is cooled until elimination of solid paraffin. Subsequent melting by heating up to a stated temperature affects differently various petroleum products; it may result in a fresh superheating and thus considerably lower the freezing-point. It is therefore imperative to subject every individual product to a series of heating tests until such temperature is reached as is *just sufficient* to liquify it anew. Subsequent cooling down to loss of mobility gives the maximum and therefore the true freezing-point.

REFINERY METHODS OF LOWERING FREEZING-POINT OF PARAFFIN FUEL. By Eng. I. O. Luchinski.

Owing to the propensity of the petroleum paraffinoid residue (mazout) to loose its fluidity, its use as fuel, and its transportation either in tank cars or by pipe-lines is very difficult. In order to obviate these difficulties, paraffinoid petroleum products are usually diluted with non-paraffinoid petroleum products, both light (kerosene and ligroine fractions) and dark ones (crude petroleum). The addition of light products affects the freezing-point but slightly. Dark petroleum products containing bituminous compounds lower the freezing-point considerably, and the mixtures made under refinery conditions show lower freezing-points than those carefully prepared in laboratories, when superheating is sedulously avoided. The lowering of the freezing-point is due to the loss, fluidity being delayed by the superheating of the mixtures and the presence of bitumens.

THE TREATMENT OF SURAKHANY AND GROZNY PARAFFINOID PETROLEUMS. By Prof. L. G. Gurvitch.

It has, so far, been impossible to deparaffinise the Grozny and Surakhany paraffinoid petroleums at the existing paraffin scale refineries, in such a manner as to make practicable to utilise the paraffin, as the latter separates in minutely-crystallised particles, which cannot be squeezed out with filter-presses. These petroleums should be subjected to a fairly strong destructive distillation, resulting in the separation of paraffinoid oils, which freeze into crystals of greater size. As to the cracking process, it is attended by numerous difficulties due to its slowness, large amounts of fuel required and the impossibility to make the distillation continuous. The principle underlying the new method of treatment is this, that the decomposition of the constituents of petroleum is much easier in a vaporised than in a liquid state. The vapours of the distillates, when issuing from the still and before entering the cooler, are heated up to 500° in a tubular superheater. Grozny mazout (residue), which has a freezing-point of 30°, when treated in this way in the laboratory still distilled 73% (freezing point of distillate, 3°). Specific weight of distillate, 0·84; viscosity low. In freezing the paraffin scale is separated in large crystals. Anticipated yield of paraffin, 1–1·5% of crude petroleum used. Residue in the still-pitch melting at 78°.

The deparaffination of Surakhany petroleum is conducted at lower superheating temperatures. This method admits continuous distillation. At present, experiments are being conducted on a practical scale.

EXTRACTION OF VASELINE FROM GROZNY PARAFFINOID-PETROLEUM. By Eng. M. B. Hazanovitch.

The paraffinoid-petroleum residue (mazout) is dissolved in light benzine (spec. wt. abt. 0·700) and treated with a mixture of petroleum soot and bone charcoal, and thoroughly mixed. After filtering through bone

charcoal the benzine is distilled with steam. The residue freezes into a yellowish vaseline of a butter-like consistency, fit for medicinal use.

In order to obtain a pure white product, the petroleum residue should be treated with fuming sulphuric acid and subsequently by alkali.

THE NECESSITY FOR CONSTRUCTION OF A PARAFFIN SCALE REFINERY AT GROZNY. By Eng. I. I. Yelin.

The increasing production of paraffinoid-petroleum, both at Grozny and Baku, brings to the fore the necessity of deparaffination, especially as regards Grozny petroleums which contain as much as 10–11% paraffin scale, the residue (mazout) having a freezing-point of over 20°. The only way to solve the difficulty is to erect at Grozny a paraffin scale refining plant for the treatment of 80 million poods of mazout with possibilities of further extensions. It should be kept in mind that if petroleum is treated with steam, the paraffin separates in minute crystals, while the cracking process incompatible with continuous distillation, demands a large number of costly stills which it is difficult to manufacture in Russia, and also a numerous staff. The deparaffination experiments conducted by Prof. L. G. Gurvitch by superheating oil vapours therefore call for special attention. The method of treatment and the type of construction of the paraffin scale refining plant, should be chosen with the greatest circumspection, so as to enable the refinery, which will probably be the largest of its kind in the world, immediately to give satisfactory results.

THE LUBRICATING OIL PROBLEM AND ATTEMPTS TO SOLVE IT. By Eng. S. A. Vishetravski.

The writer suggests a series of measures to alleviate the dearth of lubricating oils in addition to advocating the strictest economy in handling such petroleum, the majority of which is at present used as fuel. These include the use of petroleum residues (mazout), the concentration of solar oil distillates, and also as formerly suggested by the writer, the utilisation of exhauster oils which yield as much as 86% lubricants. The writer further quotes the results of technical analysis of less known petroleums, both in the Apsheron in other districts, which may be treated with a view to obtaining lubricants.

SULPHOACIDS OBTAINABLE FROM PETROLEUM HYDROCARBONS. — By G. S. Petroff.

In order to obtain sulphoacids from petroleum distillates according to Mr. Petroff's method, these should be treated with fuming sulphuric acid. The oil remaining after the elimination of acid tar is treated by alcohol or acetone. The smaller percentages of acids contained in the tar are extracted therefrom by oil, upon dilution of the tar with water. The acids belong to the saturated C_nH_{2n}–12 series. Their yield amounts to 7% of the distillate.

In technology, these sulphoacids have been termed "contact acids," by reason of their property to break up fats, for which purpose it is sufficient to use 0·5% of the reagent for 24 hours. The advantage of this method of breaking up fats are the rapidity, the simplicity, and low cost of the necessary apparatus, and the small amount of steam required.

In the textile industry "contact acids" are very useful in finishing the fabrics, also in wetting and washing underlayers, in making cotton wool, in bleaching, and in dyeing with sulphurous and substantive dyes.

The "contact" reagent has been used with great success in condensing phenols with aldehydes for the obtaining of solid condensates analogous to "Bakelites," an important point of distinction being the possibility to use no hot presses or autoclaves. The necessary amount of sulphoacids is 30% to 50%. The product obtained has been termed "carbolite." It has valuable mechanical properties, being extremely resistant to even strong acids, and, what is most important, is a powerful insulator. Solid carbolite is used for making high tension current insulators; liquid products, both for insulating and cementing purposes.

THE OXYGENATION OF PETROLEUM HYDROCARBONS AND ITS INDUSTRIAL PROSPECTS.—By B. G. Tichinin.

A survey of recent investigations of the methods of extracting acids from paraffin and other hydrocarbons, as also from residues remaining after the treatment of petroleum.

THE UTILISATION OF BY-PRODUCTS OBTAINED IN THE PROCESS OF THE AROMATISATION OF PETROLEUM.—By Eng. G. G. Godzhello.

The extraction of benzol and toluol from crude petroleum necessitated by the recent war cannot, in its present stage, compete with the extraction of these substances from coal tar. The only way to lower the cost of production so as to make competition possible, is to utilise all the by-products. It is suggested that retort-coke can be used for making electrodes and cast iron; that gas can be used as fuel; the tar, after separation of light oils, can be made to yield about 6% naphthaline; the intermediate fractions obtained in refining benzol and toluol are excellent solvents of caoutchouc. But most important of all are unsaturated compounds, including isoprene and erithrene, their contents being 0·5% and 1% respectively. In the course of refining, the majority of unsaturated compounds become concentrated into fractions boiling between 35° and 60°, and having a specific weight of about 0·775. These compounds, if subjected to prolonged settling or treated with sulphuric acid, polymerise into a viscid, mucilagenous substance soluble in benzol. The film remaining after drying of solvent is hard and flexible, and may replace oil varnish in making varnishes and colours.

EXTRACTION OF AROMATIC HYDROCARBONS FROM PETROLEUM PRODUCTS.—By G. V. Melnitzki.

The article contains an historical account of the production of benzol and toluol from petroleum, beginning from its inception. This is followed by a description of various plants used for either extracting the above products from crude petroleum or for obtaining them by the pyrogenetic decomposition of petroleum products.

TREATMENT OF PETROLEUM WITH SPECIAL REFERENCE TO GROZNY CONDITIONS.—By M. Markovitch.

The method suggested by the writer is based on M. M. Tichwinsky's Patent (Russian Patent No. 28,993 of Jan. 31st, 1917) for the extraction of benzine from crude petroleum by blowing through inert gas, from which benzine is obtained by either compression or condensation, the latent heat of liquefied benzine being utilised for heating petroleum. The method is suggested for cases where, as in the case of Grozny petroleum, the separation of a small amount of benzine and kerosene calls for an unnecessarily large amount of heat, so as to bring the total of the petroleum treated up to boiling-point.

The method has been worked out for the purpose of debenzination of petroleum in the field, so as to avoid the loss of the lighter constituents while in storage, and also for refinery treatment to obtain a yield of up to 10%.

PARAFFINOID FUEL ON RUSSIAN RAILROADS.—By Prof. A. W. Sapojnikoff. (*P.S.J.*, 1922, No. 1, p. 71.)

One of the serious difficulties for a wide application of liquid fuel on Russian railroads consists in the high percentage of paraffin scale in mazut obtained in the Grozny district (also to a less extent in Baku mazut) such mazut has a high solidifying point, which reaches occasionally 15°–25° C. for Grozny mazut and 10°–5° C. for Baku mazut. The present output at Grozny includes 70% of paraffinous oil, and only 30% of oil without paraffin scale; at Baku about 30% of paraffinous oil, but including 45% of petroleum not suitable for the manufacture of mazut.

Owing to the considerable paraffin scale content and the high solidification temperature of paraffinoid mazut all the work of its transportation, pumping and application on the locomotives is very difficult, as during the winter, even in Middle Russia, the combustible solidifies with great facility to a dense vaseline-like mass, having no fluidity. The Vladikavkaz railroad had earlier than the other railways to do with this paraffinic combustible, and has long ago adopted two practical measures for counteracting the paraffin scale content of mazut. First, Grozny prepared for this railroad during the winter months a special "mixed" combustible, with the point of solidification lowered down to 0° or even –5° C.; secondly, a whole system of preheating apparatus is introduced by this railroad in conjunction with cisterns, tanks, of tenders, and other reservoirs. Owing to the comparatively mild climate of the region traversed by the Vladikavkaz railroad these measures adopted by the railroad have proved up to now sufficient to ensure an uninterrupted use of oil combustible from Grozny all the year round.

A committee appointed to investigate the extracting of scale paraffin from petroleum recommended :—

1. The best method for lowering the solidification point of mazut consists in the preparation of "mixed" fuel from paraffinic and paraffinless masuts (or by distillation of a mixture of paraffinic and paraffinless petroleum).

2. As one of the measures of combating the paraffin scale content can be proposed the preparation of "colloidal" combustible with an admixture of coal dust; this point is being specially investigated.

THE GROZNY PARAFFINOID CRUDE OIL, AND SOME PROBLEMS OF ITS TREATMENT.—By Prof. M. M. Tichvinsky. (*P.S.J.*, 1921, No. 1–4, p. 52.)

In 1917 the production of crude oil in Grozny amounted to 94·8 mill. poods, but it would no doubt have largely exceeded 123 millions poods, but for the fire that destroyed the new oilfields. In classifying the production according to areas and qualities we obtain the following results :—

	Non-paraffinoid Oil	Paraffinoid Oil.
Old Area	60 mill. poods	12 mill. poods
New Area	3 „ „	48 „ „
	63 mill. poods	60 mill. poods

Deducting the crude oil sold or used as fuel at the fields, the following quantities are available for treating at refineries :—

	Non-Paraffinoid Oil.	Paraffinoid Oil.
Old Area	52·4 mill. poods	9·7 mill. poods
New Area	2·82 „ „	45·1 „ „
	55·22 mill. poods	54·8 mill. poods

The cold test is of –17° C. for non-paraffinoid oil, and + 5° C. and over for paraffinoid oil.

The Grozny refineries can only deal with non-paraffinoid oil, having no plant for the extraction of paraffin scale. The same plant has therefore to be used for the distillation of both kinds of oil. Non-paraffinoid oil yields 11% naphtha and 8·5% burning oil ; paraffinoid oil, 9% naphtha and 0·5% burning oil, the residue from paraffinoid oil showing a cold test of + 12° C. For the purpose of converting this residue into liquid fuel it was mixed with 15% burning oil distillate for the Caucasus region, and with 25% burning oil distillate for the South of Russia. In view, however, of a shortage of burning oil, the mixture was prepared as follows :—

50% paraffinoid oil residue .. .. ..
35% non-paraffinoid oil residue .. .. } cold test —8° C.
15% burning oil distillate .. .. ..

In some cases, a mixture of both kinds of oils (1 : 1) was distilled, sometimes even with the addition of burning oil (50 : 35 : 15). When 10–15% burning oil distillate was distilled off, the residue showed a cold test of —5° C.—8° C., but upon delivery in Russia, in winter the freezing point rose to + 5 C.

In view of the difficulties encountered in endeavouring to use as fue Grozny residue, Engineer Vishetravsky suggests that a gas plant be erected at Grozny for the transformation of the whole of paraffinoid crude oil into gas tar, which might be used as liquid fuel. Eng. Vishetravsky estimates the amount of fuel required for the purpose at 11%, the tar having a caloric power 10% below that of crude oil ; the total loss would thus represent at least 21%. A technical problem of this kind can hardly be considered rational, and thus alone would suffice for declining Mr. Vishetravsky's suggestion. What ought to be erected at Grozny is not a gas plant, but a paraffin scale plant. If, in dealing with 100 mill. poods paraffinoid crude oil containing 5% paraffin, we succeed in extracting 2%, the total yield of paraffin scale will amount to 2 million poods. That quantity will be used in the manufacture of candles required to replace stearin candles formerly produced to the amount of 1·3 million poods per annum, and which will be non-obtainable for a long time to come. The remaining 700 thousand poods would be more than sufficient to replace the 400 thousand poods of foreign paraffin scale formerly introduced into Russia every year to meet the requirements of the match manufacture, the preparation of pharmaceutical products and other industries. Pending the erection of such a plant, both the production and the treatment of paraffinoid crude oil in Grozny ought to be stopped, since the burning off paraffin scale is nothing short of squandering national wealth.

DESTRUCTIVE DISTILLATION OF THE RESIDUE FROM THE PARAFFINOID CRUDE OIL (EXPERIMENTS MADE ON THE HALL'S PLANT AT GROZNY).—By Melnizky. (*P.S.J.*, 1921, N. 1–4, p. 54.)

The residue from Grozny paraffinoid oil has the following characteristics :—

Specific gravity 0·904; cold test + 15° D. Distillation test by Engler's method.

179°–200°	6%	Specific gravity 0·845.
200°–300°	12%	
300°–350°	17%	

The residue passes through the converter tubes, the rate of feed being 360 kg. per hour, the temperature 550°–600°, and the pressure 80 lbs. The fuel consumption is 24·6 kg., *i.e.*, 6·8%.

Three different products were obtained :—

1. From the separator	65%
2. From the deflegmators (3 and 2 together)	8%
3. From the first deflegmator	10%
Gas	16%
Loss	1%

Characteristics of the Products.

Product No. 1.

Specific gravity 0·814. Cold test below —15° C. Distillation test.

92°–100°	2%	Specific gravity 0·804.
100°–125°	10%	
125°–150°	13%	
	25%	
150°–270°	38%	
270°–360°	26%	
	64%	

Product No. 2.

Specific gravity 0·840 Cold test below + 15° C. Distillation test.

144°–270°	58%	Specific gravity 0·824.
270°–360°	27%	

Product No. 3.

Specific gravity 0·900. Cold test + 15° C. Distillation test.

193°–275°	6%
275°–300°	2%
300°–320°	5%
	13%

This distillate, after having been treated with sulphuric acid and potash, was converted into a product that may successfully be used as a substitute for vaseline.

The best results were obtained at a temperature of 475°–500°, the vapours passing from the first deflegmator direct into the cooler, without entering the deflegmators No. 3 and No. 2. The distillate of vaseline obtained under these conditions had a viscosity of 4–5, a flash point of 130°–140° C., and a cold test of 29°–31° C. The residue that was simul-

taneously produced during the process was inferior in point of quality to Product No. 1, as it had a cold test not only above —2°, but usually between 0° and + 5° C.

These experiments show that by a process of destructive distillation the Grozny paraffinoid oil residue may be profitably converted into vaseline.

ON THE RELATION OF FILTRATION OF PARAFFINOUS PETROLEUM AND ITS ADSORPTION.—M. A. Raskusin. *J.R.C.S.*, Vol. 48, p. 718.

The author has made experiments in order to find whether there is any relation between the adsorption and filtration, and he found that there is no difference between those two processes. For the purposes of his experiment he took Grosny paraffinous petroleum, diluted by light benzine fractions, and the tarry matter removed by sulphuric acid. Then he passed such treated oil through a layer of fuller's earth, 25 m.m. in thickness.

The lower fractions of the solid paraffins were adsorbed in the same quantity as by ordinary filtration, and the author thinks that by increasing the thickness of the adsorber it in no way affects the results, and does not improve the quality of the filtrate. He found that by using animal charcoal a much better result was received than by fuller's earth as a filtration medium.

The author comes to the following conclusions :—

I. THEORETICAL.

(*a*) The quantitative and qualitative results of adsorption depends on the character and the quality of the adsorber, but not on the method of utilisation (whether the petroleum passes through the adsorber or is agitated with it, etc.).

(*b*) The speed of adsorption is very great, and therefore a medium of 25 m.m. in thickness is sufficient.

(*c*) The adsorption of the carbon matters in petroleum is unrecoverable

(*d*) Hot water does not extract oily and other matters from the adsorbers.

II. PRACTICAL.

(*a*) Filtration through the adsorbers has to be preferably adopted against other methods of adsorption, which demand more power and time.

(*b*) Regeneration of the adsorption after petroleum has been filtrated through ; it can only take place by calcination.

III. CHEMICAL GEOLOGICAL.

(*a*) Natural processes of filter-distillation of petroleum undoubtedly are connected with an unrecoverable process of adsorption.

(*b*) This explains why benzine does not dissolve bituminous matter from natural bitumens and other petroleum species.

(*c*) The quantitative extraction of bituminous matter from natural bitumens is made by means of CS_2 chloroform, turpentine, etc.

(*d*) The high carbon constant which indicates the character of the respective nature of petroleum at the same time indicates the amorphous character of the strata through which petroleum has passed.

(*e*) By simple filtration the natural petroleum having a carbon constant of about 18% is decolorised.

REVIEWS

"REVIEW OF BAKU PETROLEUM INDUSTRY FROM 1920–1922." Published by the Baku Government Petroleum Association.

This publication is one of the most remarkable issues on the whole petroleum question at Baku, giving complete data of the results of the nationalisation of every branch of the Petroleum Industry. The geological data on the whole Baku district is of such value that we consider an abstract would hardly do justice. We will therefore at present confine the abstracts to other branches of this industry.

EXPLOITATION OF THE PETROLEUM DISTRICT.

The production of Petroleum in Baku was in 1917 391,000,000 poods, and the amount of feet bored was 227,000. For two years the quantity of crude oil produced was reduced to about 319,049,000 poods, or one-third of the 1917 production. The number of producing wells in April, 1922, was 1,573, and new borings at about an average of only 80,000 feet per annum. The Rotary boring rigs to the extent of twenty-two in number were also introduced, and fifty-three Baku rigs were in use.

The average number of wells in exploitation for two years was :—

Balers	..	794	wells producing each	244	poods per day.
Compressors		80	" " "	2,061	" "
Spouters	..	19	" " "	3,759	" "

In the course of two years a large number of boilers were substituted by electricity ; 68·8% of oil was produced by electrical power ; 12·7% by steam, and 18·5% by internal combustion engines. (This does not include spouters.)

The production in various fields was as follows (in 1,000 poods) :—

1. Surachany field	101,535	..	31·8%
2. Bibi-Eibat (including Holy Island)	60,740	..	19·3%
3. Romany field	57,614	..	18·0%
4. Balachany field	38,592	..	12·8%
5. Sabunchy field	37,501	..	11·7%
6. Binigady field (including Shaban)	23,966	..	7·1%
	319,048	..	100%

In addition to crude oil natural gas was produced, equal in its calorific value to 3,445,000 poods of crude oil, chiefly produced from the Surachany field.

Very interesting data is given on the quantity of fuel used in the oil fields (which was for two years 18·8%). In the wells exploited by steam 26% of fuel is used ; by electricity, 5·3% ; by internal combustion engine, 3·75%.

REFINING OF PETROLEUM IN BAKU.

At the time of nationalisation there were eighteen refineries, of which at present only five are working. The capacity of those works has been enormously reduced by the absence of the materials necessary to keep them in proper repair ; in addition to this, the absence of the chemicals necessary for the treatment of kerosene and lubricating oil distillates.

For example, there is only one sulphuric acid works in action, where sulphuric anhydrate is produced by contact system ; but through the absence of platinised asbestos even this could not be worked to its full extent.

The factories associated with the treatment of crude oil are sub-divided into the following sections :—

1. Distilling.
2. Chemical.
3. Toluol.
4. Resinal.
5. Oxygen-carbide.
6. Barrel factory.

In the distilling section in the course of two years the following distillates were received :—

Gasoline	3,059,117	poods.
Lamp oil	53,383,614	,,
Solar oils	3,070,941	,,
Lubricating—		
Ordinary	12,655,003	,,
Special	126,085	,,
"Negrol"	364,062	,,
Residue	147,776,388	,,
Total	220,445,205	,,
Loss	2,337,665	,,
Total quantity treated in two years	222,782,870	,,

The works for refining and extracting paraffin scale from Cheleken crude has not been working, for such crude has not been delivered. Although there are in Baku four sulphuric acid works, only one has been in actual use, and 521,891 poods of sulphuric acid have been produced.

As regards caustic soda, three works were in operation for the purpose of caustination of soda by means of burnt lime, and 40,642 poods of caustic soda have been prepared in this way.

Utilisation of the soda by-products for the purpose of manufacturing naphtha soaps has been carried on very successfully, and 236,700 poods have been received.

Toluol factories.—There are four in Baku, but only two were in use. At Nobel's, as material, de-benzinised crude oil and solar oil were used, while at the other factory (Vopron's) kerosene distillates were in use.

For the purpose of pyrogenisation the following were used :—

De-benzinised crude	250,841	poods.
Solar oil	97,514	,,
Kerosene distillates	463,811	,,

From the above the following products were received :—

Toluene	13,708	poods.
Benzene	15,612	,,
Coke	4,566	,,

Resinal factories.—These factories have been chiefly occupied in producing a varnish on the average 1,000 poods per month. At present it has only produced 2,906 poods and resinol 3,503.

Oxygen-carbide works.—The quantity of oxygen produced in the course of two years, 31,674 c.m. The quantity of carbide, 150 poods per month.

Barrel factories.—In these two years 13,031 new barrels were made and 28,000 were repaired.

Electrification of the refineries.—In the few refineries now worked by the Government most of the steam engines have been superseded by electrical ones.

Laboratory Experiments.

The increased production of paraffinous petroleum during the last two years in the Grosny and Surachany fields has turned one's attention to the research of proper methods for the treatment of crude oil. It was evident that, under present conditions, to construct works to extract paraffin scale from 100,000,000 poods of crude per year was impossible, and therefore experiments were carried out on a technical scale according to the methods suggested by Prof. Gurvitch, by superheating the distillate vapours. The result was very successful, as the freezing-point of the residues received was from 18%—20% lower than the residue obtained by ordinary distillation.

A series of experiments were made on a practical scale for the utilisation of petroleum residues to be used as lubricants. For that purpose, residue of Balachany crude has been treated with 10% of sulphuric acid, and the products received have been experimented on an engine in the course of six weeks. The result was very satisfactory, and there was no difference between the action of this oil and the ordinary lubricating oil. Experiments were carried out in the same direction with a view to utilising solar oils for the purpose of turning them into lubricants by means of concentration. The results were successful, as the heavy solar oil from Balachany and Bibi-Eibat crude gave 15% of lubricating oil with a viscosity of 6·5 at 50°. A very interesting investigation has been made as regards crude from the Maikop district (Kalujsky), which gave 32% of lubricating distillate. The laboratory has also carried out experiments with a view to economising caustic soda at the refineries. By treating with a smaller quantity of caustic soda and then finishing with lime, thus the quantity of caustic soda used was reduced by 50%. Methods have also been worked out for preparing cylinder oils with a flash-point 350° C.

The soda by-products from lubricating oil was utilised for the purpose of destroying the emulsified petroleum from the Bibi-Eibat fields, which generally contains 22% of water and mud. The addition of 1¾% of the same by-products was enough to destroy the emulsion.

One apparatus was constructed and a series of experiments were made on the chlorinisation of natural gas, with the object of producing chloroform, and the experiments gave good results. In addition to these a number of other important investigations were carried out at the central government laboratory at Baku.

In January, 1922, there was held in Moscow the First All-Russian Congress of Oil Operators. The Members of the Congress were segregated into eleven groups. The transactions of one of these sections, the Geological Section, are now submitted to the reader. The meetings of the section were held under the permanent Chairmanship and guidance of Mr. A. Kossyguin, Mr. Majewski being Secretary. The papers read at the meetings and followed by animated discussions, fall naturally into three categories. The first category covers the papers relating to the penetration of water into the producing strata of the Baku and Grozny oil-bearing districts. The second category comprises questions of organisation, and more especially the establishment and working of geological survey sections, which have recently been formed in the Baku and Grozny districts. The papers comprised in the third category deal with future prospects of petroleum industry as determined by the reserve fund of oil-bearing lands available, and the programme of work for the coming quinquennial period.

1. Water troubles in oil-bearing districts. This subject has been dealt with in eleven papers.

D. Golubiatnikoff, in speaking of underground waters in the Baku district recalls the fact that these waters are brines, the principal constituent of which is NaCl, and which are chiefly characterised by the almost total absence of sulphates. The state of mineralisation of these waters is, as a rule, in inverse ratio to their stratigraphical depth. The waters which find their way into the oil wells may be of threefold origin. They are either waters from the sand that is being worked for petroleum, or overhead waters from overlying horizons, or bottom waters entering the sand from the lower levels along joint planes. The origin of water in a well may be ascertained by geothermic observations. If the temperature of water is equal to the temperature at the bottom of the well, calculated by the thermal gradient of any given area (*e.g.*, 11·7 sazh. for Bibi-Eybat, 11·5 sazh for Surakhany, etc.), it originates from the stratum drilled: if lower, the water is overhead water, while bottom waters have a higher temperature than that at the bottom of the well.

Mr. M. Abramovitch, Chief of the "Aznepht" (Baku) Geological Survey, gives in his paper an outline of the position in regard to the penetration of water into the producing areas of Baku district, and suggests some measures to counteract same. The study of this question should begin with investigations as to the occurrence of underground waters and their influence on the flow of water in a well. With this end in view all wells are now being systematically tested. Water sands ought to be divided into unquestionably primary water sands, and secondary water sands (*i.e.*, water-logged oil sand). The difference in the encroachment of water in various localities is due to the difference in the rate of oil production (*i.e.*, it is determined by the volume of liquid yielded by one well in a day), and the percentage of water in the liquid yielded. These data have been figured out for each locality, and are embodied in 7 tables appended to the paper. In the wells which are now being drilled every effort is made to exclude water, and all the wells unfit for further production should be abandoned, those which offer the greatest danger of the encroachment of oil-bearing strata by water being shut down first.

Mr. I. Samoiloff's paper deals with the penetration of water into the oil-bearing strata of Balakhany district. The water-logging of oil sands above the so-called "interpretation" does not give rise to any serious apprehensions as regards the spread of the water trouble, as in these horizons a certain "*statu quo*" has been established. Some light is thrown on this question by the tables appended to the paper, which contain preliminary approximate calculations relating to this matter. Penetration of water is, of course, already going on, but on a small scale; it mainly affects the 4th and 5th horizons and is hardly noticeable in the others, and is not threatening in character. This, however, does not apply to the Kirmaki series, which lies below the "interruption," and has been invaded by waters from the thick group of sands which constitute the "interruption." Many of the wells which have penetrated the argillaceous sandy roof of the Kirmaki series had to be shut down owing to the fact that overhead waters had found their way into the bottom of the well. The time has come to consider the question of abandoning the wells which give access to water into the Kirmaki series.

Mr. Appresoff's paper on water troubles in Surakhany district deals with the stratigraphy of the eight water sands, and contains general suggestions as to the prevention of water troubles in various localities of the district.

The next following article contains instructions as to the working out of sections, and the description of the life of each well being a necessary condition to the two foregoing reports.

The concluding chapter of this section contains the debates on the subject of the report submitted by the "Aznepht" Geological Survey. These debates are almost exclusively confined to various technical points in connexion with the carrying out of the programme of testing oil wells.

III. The third category comprises nine papers, and deals with the future prospects of petroleum industry, as determined by the reserve funds of oil-bearing lands available, and a programme of work for the coming quinquennial period.

Somewhat apart in this group stands Mr. S. Charnotski's report on "The Method of Estimating the Reserves of Oil in Oilfields, and an Attempt to Apply it to the Maikop and Grozny Districts." The Russian text contains but a summary of that paper. The Report itself, having already been published in book-form by the editors of the Review, *Petroleum and Shale Industry*.

The remaining papers of the third category keep strictly within the limits of their subjects. They give a detailed estimate of the total reserves of oil-bearing lands available, and discuss the future possibilities of development of the Russian petroleum industry.

Mr. S. Mironoff's paper deals with the Ural region which is an extremely extensive oil-bearing district. Indications of oil have been recorded over an area of about 70,000 square versts. Out of the numerous oil fields in this district the following three have been tested more or less satisfactorily by deep boring; Dossor, Makat, and Novo-Bogatynsk. Dossor has yielded, within a brief period of time, over 80 million poods of oil. Makat will yield several million poods a year, if operations are conducted with the requisite degree of efficiency. Novo-Bogatynsk oil field calls for further steady and persevering investigations. As regards further testing of the territory, it is imperative in the first place to drill shallow wells into the Blauli oil field, to make a preliminary test at Kara-Chungul, and to complete the well commenced at Mortuk.

The Ural district is also dealt with in Mr. Tikhonovitch's paper, who is of opinion that comparatively small disbursements on testing the territory would suffice to elucidate the commercial importance of Karaton and Kara-Chungul, but considerable expenditure would be involved in testing the Bek-Beke, Akat-Kul, and Satep-Alta oil fields. Mr. Tikhonovitch also insists on the necessity of the well drilled into Mortuk oil field, which has been stopped practically on the head of the Dossor series; he also recommends that a well be drilled into the Dunguluk-Sore oil field.

In his paper on the Terek oil fields Mr. P. Prokopoff suggests the following programme of work to further the development of petroleum industry. In Old Grozny district, drilling in area around Solanaya Balka, and on its extension to the south-east. Also, test wells to be drilled into the sands underlying the 16th and 17th strata. In New Grozny district, the production should also be increased by tapping the lower sands, and by extending the present producing area to eastward and westward. In the Black Mountians (Chorny Gori) new areas may be mapped out in the region around Miatly and Beroy. In the Sundja ridge borings should be made at the western and eastern extremities. Terek Ridge not only needs further development, but also calls for a study of its complicated geological structure.

Another paper by Mr. Prokopoff deals with the Kuban oil-bearing district. As regards the Maikop oil fields of light oil, the investigations so successfully begun by Mr. I. Goubkin should be continued further to the north-west so as to follow in that direction the strike of the peculiar sleeve-shaped body of light oil. Another step towards the investigation of the Maikop district is the delineation of the boundaries of the oilfields containing heavy oil. The northern portion of Hadizhenski area should be further tested in spite of the fact that some of the wells have already reached a considerable depth (2500 to 3000 ft.). The region around Kaluzhskaya should be made the object of a systematic geological survey, combined with the drilling of shallow wells for purely testing purposes. A similar detailed geological survey is needed by the Jll district In the Krimski area it might prove suggestive to dig pits and drill shallow boreholes into the dome of the anticline which runs in this locality.

Mr. K. Kalitski's paper deals with future prospects of petroleum and ozokerite production in the Transcaspian territories. The oilfields of these territories, represented by Cheleken Island, Naphtedag and Boyadag, have been brought up to the surface by underground intrusions. But for these intrusions the oil-bearing series would lie quietly deep below the Caspian plain, without betraying the presence of any surface indication. As there is no casual connexion between underground intrusions and the occurrence of oil, the fact of petroleum being found in the island of Cheleken, at Nephtedag and Boyadag, cannot be accounted for by an underground intrusion. This is but a fortuitous coincidence, as is amply proved by the case of Mount Monzhukla, where we find a beautiful dome structure fractured by great failt fissures, yet absolutely barren of oil indications. Here, on Mount Monzhukla, we also have an underground intrusion, but this occurred in a locality barren of oil. In the island of Cheleken, Nephtedag, and Boyadag, the intrusion occurred below oilfields and dislocated them partially or entirely. It is quite obvious that over the area of the Caspian plain, roughly delineated in the north and east by lines connecting Cheleken, Nephtedag and Boyadag, and the Keimir water wells, and in the west by the coast of the Caspian Sea, vast supplies of petroleum must lie concealed in the depths, without betraying their presence by any surface indications, as they are overlaid by great thicknesses of Pliocene clays. The Transcaspian territories thus contain vast reserves of oil-bearing lands available for future times, and which cannot, of course, be drilled under present conditions. Entirely different is the case of ozokerite. As this mineral is only associated with strongly dislocated paraffin oilfields (ozokerite veins) or with outcrops of strata containing oil of paraffin base (stratum ozokerite), no fresh discoveries of ozokerite producing areas, besides those already known in the islands of Cheleken and Nephtedag, can be expected within the Transcaspian territory.

Another paper by Mr. Kalitski deals with the prospects of petroleum and ozokerite production in the Ferghana region. In this district it may prove most suggestive to drill test wells deep into the Kapchagai anticlinal fold running at a verst to the north from the well-known Chimion flexure and parallel to it, and a narrow dome structure one or two versts to the westward from Shursu oilfield. Quite untouched by drilling is the Tash-ra-vat oilfield in the Samarkand district, which is a natural extension to the westward of the southern belt of the Ferghana oilfield. As regards ozokerite there is in addition to the well-known beds at Shursu and Selrokho, a third and absolutely intact bed, which, to all appearances, is very similar to that of Selrokho.

A third paper by Mr. Kalitski deals with the prospects of oil production in the Ukhta region. Here we have a vast area of about 1200 square versts, which is considered oil-bearing. Numerous bore-holes have shown that oil-bearing sands are restricted in number, that these sands are not very thick and give but a limited yield of oil. The best among the producing wells has yielded, during the period of test drilling, about 3000 poods of oil. The whole of the oil-bearing series of the Ukhta region has been pierced by a number of deep wells, which have penetrated down to the basal beds of sericitic shists that underlie this oilfield.

A fourth paper by Mr. Kalitski deals with the Volga region. At more than ten localities tar sands have been proved in this vast region, but only the one at Shuguri is being worked. No liquid oil has, so far, been discovered in this region. As all hopes to strike oil are only based on the possibility of discovering tar sands which do not crop out at the surface, and have therefore escaped dedunation, it would be well to drill a well in the centre of the area bound up in the north and in the east by Lesnaya Shesma, and in the west and south by Stepnaya Shesma—*i.e.*, somewhere around Skhodnevo or Nazarovka. This space seems to be encased on three sides by outcrops of tar sands. A well drilled in the vicinity of Skhodnevo or Nazarovka, and reaching down into the Carboniferous formation, might reveal strata of tar sands hidden deep below; there is, therefore, some hope of striking oil. Since such drilling would be conducted at random and may lead to entirely unsatisfactory results, it would be preferable to locate the well in the vicinity of Staro-Semenkino, where it is sure to strike at least one tar sands, thus revealing what its oil contents are likely to be at a great distance from its natural outcrops.

The general conclusion to be drawn from all the papers of the third category is this, that as regards the reserve fund of oil-bearing lands and the prospects of petroleum production in Russia, the possibilities of future development are entirely satisfactory.

Shales and Phosphorites in East of the Volga River and "Syrt" Region.—A. Rozanov. *P.S.J.*, 1921, Nos. 1–4, p. 155.

The author visited during the summer of 1920 oil-shale deposits situated in the vicinity of Kashpoor (Sysran district), in some places east of the Volga (Samara province), and in the northern part of the Simbirsk province. His purpose was to study the possibility of a combined mining of oil-shales and phosphorites.

The results of the observations made show the following:—

In the vicinity of Kashpoor, and on the Volga-Sviajsk watershed—in the northern part of the Simbirsk province—a combined production of both minerals must be considered as impossible by means of underground mining because of technical difficulties; the necessity of breaking a hard roofing, a very great compactness of the phosphorite layer; the necessity after the elevation of the drift-way's level to abandon the exploitation of some shale-layers. On the contrary, a combined production of these two minerals by means of open works is quite possible and rational, but unfortunately the area of the ground on which such an exploitation is possible is in both districts limited.

In the "Syrt" region a close disposition of the oil-shale and phosphorite horizon which could possess a practical significance cannot be observed.

It must be said that the question of a combined production of oil-shales and phosphorites is wider and more complicated than it seems, because (1) in the region of the Soorsk-Sviajsk watershed the relation between the lower-neocomian phosphorite horizon and the Lower Volga oil-shales permits theoretically the possibility of underground works;

(2) there exists a series of districts where the principal useful minerals are not the oil-shales but the phosphorite, and where the former can be mined from underground works simultaneously with the phosphorite (Kostroma and Moscow districts). In addition to this in the basin of the Piany River there exists a place where the Lower-Neocomian phosphorite and the relevant Lower Volga can be mined together by open works.

EXAMINATION OF VOLGA SHALE.—L. Koorbatov. *P.S.J.*, 1921, Nos. 1–4, p. 160.

Shale from Under (Volga) contained :—

Water	28%
Volatile Matter	24·3%
Coke and Ash	46·9%
	99·2%
Ash	71·5%
Carbon	16·32%

Heated at 330° for 28 hours, 5·3% of tar with a sp. gr. of 9·987, containing 5·5% sulphur.

For the extraction of the nitrogenous basic matter, 7·125 kg. of crude oil were taken, which gave 132 gr. of organic bases. After the removing of the primary and secondary bases by means of diazotroping the tertiary bases were fractionated ; 47·8 gr. being distilled over before 220°.

The following substances in the shape of picric acid salts were obtained from the different fractions ; pyridin, α-picolin, β-picolin, γ-picolin and x-α-lytidin.

The presence of the β-picolin in the crude oil permits a possibility of a part of the oil-shale organic matter being formed by fats.

The extraction of the oil-shale's organic matter by means of ordinary dissolvents such as ether, benzol and aceton gave but little result. A very small quantity of waxy matter can be extracted by benzol. Comparatively lightly the organic matter can be extracted by means of alkalies—up to 32%—from where it can be got out by mineral acid in the shape of an amorphous mass. The filtrated, washed and dried brownish-black mass was analysed and showed the following structures :—

C = 57·8%
H = 4·7%
O + N = 37·5%
S = traces.

These preliminary investigations permit one to refer the organic substance of the Volga oil-shales to the " Gumin matter," from which, judging by American experiments, fats had already been extracted.

THE EXTRACTION OF BENZOL AND TOLUOL FROM NAPHTHA BY THE PYROGENETIC METHOD.—A. L. Pokrovski. *P.S.J.*, 1921, Nos. 5–8, p. 83.

The process may be roughly sub-divided into three stages :—

1. The pyrogenetic treatment of naphtha, or of its oily distillates.
2. The extraction of " light oil " from the products of pyrogenesis (tar and scrubber oil), and preparing the same for rectification.
3. The rectification and the extraction of benzol and toluol.

In the *First Stage*, either retort furnaces or generators are used. The purpose of either is to collect the products of decomposition which are successively passed through the hydraulic main, the air-cooling cylinders, the water coils and the scrubbers. The difference between these two alternative appliances lies in their heating apparatus and the efficiency

of its component parts. The retort furnaces are continually working, slightly inclined cast-iron cylinders some three metres long, heated by an outside flame. The pinch generators are periodically working appliances (iron cylinders 5·85 metres high, and 2·16 metres in diameter, provided with a made-up body lining). They are connected in pairs, and the naphtha is distilled therein by the heat accumulated through their having been previously heated by oil burners. Every retort has a capacity of about 21 poods, and every generator a capacity of 1100 poods per 24 h. In either case the temperature of pyrogenesis is 680–700° C., and the process is conducted in vacuum.

The *Second Stage* of the process is made up of a series of auxiliary operations, viz. :—

1. The distillation of tar in an ordinary petroleum still over an open fire, the following fractions being recovered : Light oil, up to 200° C. ; naphthaline oil from 200° to 225° C. ; and scrubber oil from 225° to 275° C. The latter absorbs the remainder of the hydrocarbon vapours circulated throughout the cooling apparatus. It is then regenerated in the still over an open fire and may be used over again ; the light oil obtained therefrom (up to 200° C.) is mixed with that extracted from the tar, and the mixture is used for

2. Distillation in combination with water vapours in an ordinary petroleum still. About two-thirds of the total volume of the product is lost by weathering, thus freeing the material under treatment from any unnecessary residue containing no benzine and but a slight percentage of toluol (not over 9·3%) ; this intensifies the concentration of the final products.

3. The intensification of concentration referred to above is, to a great extent (56% as against 28% in light oil), achieved by previous rectification in ordinary spirit distilling towers. The separation of the fractions takes place within narrow limits, and is characterised by the following figures :—

Top, recoverable up to	..	73° C.	to the extent of	8·2%
Light benzol recoverable from	73°–	79° C.	„ „ „	4·7%
Benzol fraction	79°–	83° C.	„ „ „	33·3%
Benzol-toluol fraction ..	83°–	108°C.	„ „ „	6·8%
Toluol „ ..	108°–	113°C.	„ „ „	28·4%
Toluol-xylol „ ..	113°–	122°C.	„ „ „	1·1%
Residue in still				14·0%
Loss				3·5%
			Total	100·0%

The basic fractions, *i.e.*, the benzol and the toluol fractions, are subjected to a process of chemical refining, and all the intermediary fractions (with the exception of the bottom fractions) are collected in a separate vessel and rectified anew before refining. The top and the bottom fractions, the residue in the still, as also the intermediate fractions resulting from a reiterated preliminary rectification have, up to the present, been used as fuel. To give an idea of the quantitative proportions of these fractions, the following statement may be made : although widely different amounts of benzol and of toluol are obtained from tar (32% and about 1%, respectively), while scrubber oil contains practically an equal percentage of these constituents (12·8% and 13·6%, respectively), this difference is eventually wiped out, owing to the fact that tar yields about six times as much light oil as scrubber oil does. Thus the ratio of benzol is in round figures, equal to 3 : 2.

4. The chemical refining, applied to every basic fraction separately, consists in the treatment of the product with sulphuric acid up to the extent of 23% of the weight of the distillate, the acid being added in small quantities, so as to avoid any too violent reaction. With the same object in view, the refining apparatus—an iron cylindrical vessel with a conical bottom holding 700 poods of the product—is provided with a water jacket. Thereafter the distillate is washed in water and then treated anew with 1% solution of potassium manganate and then washed again. Benzol should be treated with 98% sulphuric acid, but a 96% one is preferable for toluol, so as to avoid any material loss by sulphuration. The product is subsequently admitted into a similar apparatus placed below the former one, for the purpose of being treated with a solution of caustic soda (3° Bé) used to the extent of 3% of the distillate by weight. This is followed by a last washing in water. Every one of these successive washings requires an amount of water equal to about 10% of the product by weight. The duration of the whole process of chemical refining is of 60 to 16 hours, according to the number of sulphuric acid charges. The heterogeneity of the medium is avoided by an air-blast forced into the lower part of the apparatus. A standard of the adequacy of refining is supplied by the bromic number, which, in compliance with official requirements, should not exceed 1.

5. Upon completion of chemical refining, the product is again subjected to distillation with water vapours, for the purpose, as in the first instance (*cf.* par. 2 of the second stage) of freeing it from unnecessary ballast, and also of tying up the sulphuric gas, failing which no rectification would be possible. With this object in view 1% of caustic soda (3° Bé) is introduced into the still. The recovery of distillate takes place up to 100° C., the percentage usually collected being about 75% of each charge. The fractions recovered between 100° and 102° C. are separated from the rest, and subjected to a renewed refining and redistillation by water vapours. The fraction is usually collected at the rate of 7% of the charge; it is inferior to the first fraction in point of quality, its bromic number being 1·5, as against 1·0. The benzol and the toluol fractions are then subjected to

The *Third Operation—i.e.*, the final rectification in Savalle's spirit distilling towers. The benzol fraction is charged first. It gives :—

Light benzol, about	8%
Pure „ „	80%
(boiling point 79·3–80·5° C.)	
Residue in still „	8·5%
Loss	3·5%
	100·0%

The toluol fraction is charged over the residue, and is decomposed into :—

Light benzol, about	2%
Pure „ „	9%
Benzol toluol „	6%
Pure toluol „	65%
(boiling point 109·2–110·2° C.)	
Residue in still, about	16%
Loss „	2%
	100%

Most important in the refining process are the moments of transition from the separation of pure basic fractions to the removal of intermediate fractions, and *vice versa*. The last-mentioned operation is carried out under the uninterrupted supervision of the laboratory, who gives directions as to such transitions being made when they are satisfied that the samples have reached the boiling-points specified above for every fraction. As to the transition from basic to intermediate fractions, it is made according to the readings of a thermometer placed on the pipe connecting the deflegmator with the cooler, the rise of temperature being in such cases rather abrupt. In effecting such transitions the very speed of the rectifying operation is altered. The maximum speed attainable on any given apparatus is used when basic fractions are being removed, and it should be reduced three or four times when collecting intermediate fractions. The difficulty of properly separating the fractions lies in a smooth successive change of speeds, which can only be achieved by maintaining at a permanent level the vapour pressure in the still (about 3 pounds) and in the coils, and the water pressure in the deflegmator. The accumulated intermediate fractions are collected separately, and subjected to a repeated rectification similar in all points to the one just described, they being fed into the still in succession, beginning from the highest product. New intermediate fractions are formed in this process, but the benzol and toluol contained therein are of so inferior a quality as to warrant no further rectification; they are therefore collected as a mixture known under the name of "solvent."

THE FLOODING OF THE BAKU OILFIELDS.—M. Smirnoff. *P.S.J.*, 1921, Nos. 1–4, p. 45.

The comparison of the exploitation data of the Baku oilfields during January, 1918, January, 1920, and August, 1920, shows a rapid flooding of these pools, caused by the lowering of the intensity of exploitation.

The results of January, 1918, and January, 1920, show that with the lessening of the number of exploited wells from 2768 to 1734, and of the absolute monthly production from 21 millions of poods to 176 millions, the middle daily yield per well had increased from 278 poods (34$\frac{3}{4}$ barrels) to 381 poods (47$\frac{5}{8}$ barrels), because only the most productive wells were pumped.

From January to August, 1920, can be noted a further decrease of the absolute production—to 10 millions, a reduction of the number of pumped wells (1112), and also a decline of the yield per well (372 poods), only the best wells being pumped.

If we make similar observations upon the different pools of the Baku district between January, 1918, and January, 1920, we shall always find the same raising of the daily production per well explained by the same cause.

Between January and August, 1920, on the "Old Pools" which possessed a greater quantity of wells, we can observe the falling of the middle daily yield per well, and the diminishing of the quantity of working wells.

The flooding of the deposits by water could be particularly noted during this period of time on the Romany and Bibi-Eibat oil pools. On the Bibi-Eibat field between the 1—111—1917 and the 1—111—1920 was noted a raising of the liquid in the wells to 33 sajenes (231 feet), caused naturally by water.

All the abovesaid shows that the flooding of the Baku oilfields is progressing rather rapidly, and that most energetic measures must be taken towards their preservation and re-establishment.

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PRODUCTION OF LIQUID HYDROCARBONS FROM CARBON OR CARBON COMPOUNDS BY MEANS OF THE ELECTRIC ARC.—K. Wilkens. Ger. Pat. 369,367. *J. Soc. Chem. Ind.* **42**, 393A.

Abstracts.

Compiled by P. Dvotkowitz, Ph.D., H. Moore, M.Sc., A.I.C., F.C.S., F. G. P. Remfry, B.A., D.Sc., A.I.C., and T. A. Smith, B.Sc., F.I.C.

General.

Oxidation of Paraffin Wax by Atmospheric Oxygen.—A. Grun and E. Ulbrich. *Z. angew. Chem.* 1923, **36**, 125–6.

By increasing the amount of air passed through melted paraffin wax from 150 to 1,200 litres per hour, other conditions being kept constant, an increase in the yield of insoluble fatty acids from 36·5% to 61·7% was obtained. The moisture content of the air also has some bearing on the yield, but the exact relationship has not yet been determined. The extra agitation by the larger volume of air, rather than the augmentation of the oxygen supply, is the chief cause of the increase in yield. It was found that even when the oxygen was reduced to 1·3% by weight by means of adding CO_2 to air a marked oxidising action occurred when the mixture was passed through paraffin wax at 160° C.

F. G. P. R.

Distribution of Sulphur in Petroleum Products.—G. Egloff and J. C. Morrell. *Chem. Met. Eng.* 1923, **28**, 14, 633–5.

Sulphur occurs in crude petroleum and its commercial cuts as free sulphur, hydrogen sulphide and organic sulphur compounds of which the following have been identified—thiophenes, hydrothiophenes, sulphonic acids, alkylsulphates, alkyl sulphides and mercaptans. In nitrogenous organic material such as albumin, gelatin, etc., Beilby has shown that distillation yields (1) ammonia, (2) oil rich in alkaloidal bodies, and (3) carbonaceous residue containing a large proportion of the original nitrogen. Oil shale, peat and coal give similar decomposition products, the nitrogen content increasing in the same order. Perkins has shown (this Journal 1917, **3**, 239) that the percentage of sulphur in an American crude oil increased with rise in boiling point. The present investigation was carried out on the primary products resulting from the cracking of a Mexican gas oil by a continuous liquid-phase process. The percentage of sulphur was found to increase with the density of the liquid and solid products, but was highest in the gas. This is closely analogous to the distribution of nitrogen in the destructive distillation of oil-shale.

F. G. P. R.

The Optical Behaviour and Some Other Properties of Egyptian Petroleum.—M. A. Rakusin. *Petroleum* 1923, 19, 412–413.

Some cargoes of Egyptian petroleum residues which arrived at St. Petersburg early in the war, being analysed, were found to have sp.gr. at 15° C. 0·916, viscosity Engler at 50° C. 4·98, solidification pt. in 15 mm. tube 18° C., in 28 mm. tube, 15° C., flash point 125° C., asphaltum content 38%, and sulphur content 1·17%. 69·3% came over up to 270° C. when distilled in Engler flask *in vacuo*. The oil was optically

alive. As the crude oil had been distilled down to coke and thick asphalt, a complete decomposition of the solid paraffins must have taken place.
H. M.

ENGINE EXPERIMENTS WITH OXIDISED FUELS.—J. H. James and F. C. Zeisenheim. *Chem. Met. Eng.* **28**, 12, 543.

Engine experiments with a fuel made by the catalytic oxidation of kerosene and a fuel of similar character made by the oxidation of gas oil show that the oxidised kerosenes develop approximately the same power as ordinary kerosene in spite of the fact that their thermal value is one-eighth less. It is believed that better combustion of these oxidised fuels takes place. The oxidised kerosenes show lower detonation tendencies than straight hydrocarbon fuel. Similar experiments with lower boiling oxidised fuels in an automobile engine gave similar results.
T. A. S.

GUM FORMATION WITH OXIDISED FUELS.—*Chem. Met. Eng.* **28**, 12,545.

Investigation shows that oxidised gasoline gives a heavy yield of gum and the question of the importance of this gum formation when the fuel is used in an internal combustion engine has been raised. The gum is in the fraction boiling over 300° C. Oxidation product fractions over 300° C, have been vacuum distilled and used as lubricating oils in a kerosene engine. There was no varnish formation with this lubricant and the carbon formation was only half that formed from well-known commercial engine oil lubricants. This carbon was of a light, fluffy character.
T. A. S.

COLLOID CHEMISTRY AND PETROLEUM INDUSTRY.—R. Koetschau. *Kolloid—Z.*, 31, 314–9 (192).

An address. The theory of colloidal dispersion of asphalt, paraffin, fluorescent substances, etc., applies to the substances generally considered to be admired or in chemical union with the oil. Many successful applications of colloidal chemical principles are given and problems indicated.
T. A. S.

HEAVY OIL ENGINE WORKING COSTS.—Report of Diesel Engine. Users' Association, March 2, 1923.

The working costs of 6 undertakings using heavy oil engines are given, the capacities varying from 100 to 600 kilowatts. The average "engine cost" per unit generated in 1917 was 0·821 pence and in 1921 1·039 pence. The lowest costs given are those of an undertaking which in 1917 generated nearly 2,000,000 kilowatts and whose costs per unit for the year were made up as follows:—

Fuel	0·441	pence.
Lubricating oil	0·036	,,
Water	0·002	,,
Stores	0·011	,,
Wages	0·107	,,
Maintenance (including repairs)	0·116	,,
		,,
Total	0·713	,,

The average cost of fuel to this undertaking in the year was 112s. per ton. The increase in cost per unit in the period under survey is mainly due to the increased cost of fuel, in one case 278s. per ton, and to increased wages. With present-day costs of labour and material 0·789

pence may be taken as the "engine cost" per unit of an installation, using Diesel engines, having an annual plant load factor of not less than 15 per cent. and an output in the neighbourhood of 1,000,000 units per annum. On Indian undertakings the costs for fuel and wages are less than in England. Owing to the poor nature of the coal available and the distance from collieries heavy oil engines would appear to have a very promising future in India. T. A. S.

SOME FACTORS AFFECTING THE UTILISATION OF HEAVY FUEL OILS IN INTERNAL-COMBUSTION ENGINES.—H. Moore. Paper read before the North East Coast Institution of Engineers and Shipbuilders, December 1, 1922.*

The difficulties in burning heavy fuel oils in heavy oil engines may be divided into three classes, namely, (1) ignition difficulties; (2) difficulties in burning the oil after ignition has taken place; and (3) difficulties caused by the presence of small quantities of impurities. Heavy oil engines may be classified in three groups, namely: (1) Diesel engines, utilising compressed air for injecting the fuel, in which ignition is obtained by the heat of compression; (2) cold-starting engines, in which the charge is fired by spontaneous ignition, and the fuel pulverised by mechanical means, without the use of blast air; and (3) hot-bulb engines, in which the fuel is injected by mechanical means, but in which external heat is applied to ensure ignition of the fuel. The capacity of an oil engine for burning heavy fuels is dependent to a marked extent upon the size, speed, compression, fuel-valve timing, etc., of the engine. In Diesel and cold-starting engines the temperature of compression must exceed the temperature of spontaneous ignition of the fuel. In burning coal-tar products difficulty is met with in starting and low load running, as the heat of compression does not sufficiently exceed the ignition temperature. There is little difference in ignition temperature among fuels of the same type, but great difference between that of petroleum oils and that of tar oils. That temperature (in oxygen) for petrol is 270°–279° C., for kerosene 251°–253° C., for residual oils 259·5°–269° C., for benzol 566° C., for creosote from gas works tars 415°–473° C., for creosote from low temperature tars 290°–350° C., and for shale oils 251° C. In the formula $T_2 - T_1\left(\frac{P_2}{P_1}\right)^{\frac{n-1}{n}}$ for temperature of compression n is approximately 1·35 for water-cooled internal-combustion engines. It is necessary to employ temperatures higher than theoretical, to ensure certain ignition under adverse conditions. Engines operating on the constant volume cycle compress an explosive mixture in the cylinder, and it is necessary to employ a much lower pressure than that of ignition in order to avoid pre-ignition. When high compressions are employed in petrol engines "pinking" results, an effect produced by the rapid propagation of the flame when the temperature approaches the ignition point. The addition of small quantities of some chemical substances has the effect of allowing the limiting compression to be raised, one substance possessing 625 times the effect of benzol in this respect. The ignition of the more refractory oils is not difficult in the hot-bulb engine, the necessary temperature being easily available. To burn oils of high ignition point on a Diesel engine pilot ignition gear has been employed,

* Members of the Institution of Petroleum Technologists can obtain copies of the ful paper, with discussion and Mr. Moore's reply, at a special price of 3s. 9d. each, on application to the Secretary of the North East Coast Institution of Engineers and Shipbuilders, Bolbec Hall, Newcastle-upon-Tyne.

a small charge of easily inflammable oil being injected prior to the main fuel charge. Two fuel pumps are employed, and the ignition oil used is 5%–10% of the total fuel. Tar oil may be burned by using a pilot charge of the same tar oil, which is not pulverised, and therefore is not cooled by the blast. A similar effect may be obtained without pilot gear by a modification of the fuel valve to allow a small advance charge to pass. Oils and tars from the low temperature distillation of coal are intermediate in ignition point between petroleum oils and ordinary tars. The burning of the oil during the expansion stroke is generally a problem separate from that of ignition, but sometimes a feeble start may influence the burning during the rest of the stroke. The result is a smoky exhaust, which may generally be remedied by raising the temperature of the circulating-water outlet. The burning of the fuel is influenced by the compression pressure, the cylinder dimensions, the presence or absence of blast air, the action of the hot bulb, the temperature of the circulating water, the degree of atomisation, the speed of the engine, and the shape of the combustion space. The oxygen, nitrogen and sulphur contents of crude petroleum oils may be of great importance, as these elements may enter into the composition of a large part of the oil. Crude oils contain solids, such as waxes and bituminous bodies in solution. The bituminous bodies cannot be distilled without being broken up. Fuel oils being frequently residual oils, the bituminous substances may be present in a greater proportion than in the crude oil. The hardness and resistance to combustion of a bitumen is allied to the difficulty of dissolving it, as in alcohol-ether mixture or petroleum spirit. Bitumens precipitated from an oil by an alcohol-ether solution are generally termed soft asphaltum, and those precipitated by petroleum spirit hard asphaltum. The former value is generally the larger, and represents the soft and the hard asphaltum taken together. The bituminous bodies readily coke in the engine, and may form deposits in the combustion space, smoky exhaust, and pitting of the exhaust valve. They increase the viscosity of the fuel, and so make it difficult to obtain efficient pulverisation. This difficulty may be to a large extent overcome by pre-heating the fuel oil; excessive heating, however, may crack the oil and form deposits, and much of the pre-heat is lost in the pulverising devices. The hard asphaltum is a most undesirable constituent of oils. Argentine oils containing over 30% soft asphaltum are not so difficult to burn as Mexican oils of much lower soft asphaltum content, but larger hard asphaltum content. The presence of bituminous substances increases the viscosity of oils greatly, but does not raise the solidification point seriously. Waxes raise the solidification point, or "cold test" of the oils. This interferes with the settling of sand and water, and paraffin wax, though perfectly combustible, may be the cause of other troubles. Petroleum distillates are free from bitumen, and are generally satisfactory fuels. Shale oils are chemically similar to many petroleum oils and are free from bitumen. Norfolk shale oils contain 6%–8% of sulphur, but this would probably not have any corrosive effect on engines, for corrosion from sulphur only takes place where the temperature falls so low as to allow of condensation, as in long exhaust pipes and silencers. The coal-tar distillates burned in heavy oil engines are free from bituminous bodies and "free carbon." Raw coal-tars are burned in Diesel engines on a large scale on the Continent. Their high free carbon content tends to cause pitting of the exhaust valves in four-stroke engines. The free carbon content of tars is determined by dissolving in a mixture of acetic acid and toluene and filtering off

the insoluble portion. Vertical-retort tar has an average content of 5·2% of free carbon, inclined-retort tar 17·0%, horizontal-retort tar 20·8%, coke-oven tar 10·4%, and low temperature carbonisation tar 1·0%. The better varieties of low temperature tar may be burned in Diesel and cold-starting engines with little difficulty. The naphthalene and anthracene present in coal tar distillates are good fuels, but may prevent the settling out of water and ash, and separate out from the oil in cold weather. Free carbon and bituminous substances may be precipitated from raw coal tars by the addition of a small quantity of petroleum oil. Water, sand and iron rust are common impurities in fuel oils. Fibrous materials are sometimes found in oils which have been delivered in barrels. Tar distillates sometimes contain sodium sulphate and sodium carbonate from their treatment in the distillery. The water content of oil is determined by distillation and the ash value by burning away the oil, when inorganic substances remain. Organic impurities such as leaves and fibre are determined by dissolving the oil and separating the residue by filtration. When water is present in fuel oil the amount present in each stroke of the fuel pump is irregular, and when the pump receives a full stroke of water the engine missfires, and is overloaded on subsequent strokes. This causes a smoky exhaust, and the lubricant is contaminated with tarry substances from the half-burned fuel oil. The presence of small quantities of ash is a serious matter, and it is usual to limit the ash content to 0·05% or 0·08%. Ash is liable to accumulate on the cylinder walls, forming an erosive paste. A measure of the erosive properties of the ash can be made by rubbing it between the finger and the thumb. The ash in Mexican oils contains vanadium oxide. Pulverisers are occasionally choked by mechanical impurities. An effective device by the author to prevent choking is illustrated. In some cases pulveriser deposits may be formed by oxidation of some constituents of the oil in the presence of high-pressure air. Water, ash and mechanical impurities may be removed from oil by allowing it to settle with or without the application of heat, or more rapidly by the use of high-speed centrifuges. The more viscous oils are generally highest in ash, water and mechanical impurities contents, high viscosity interfering with the settling-out process which takes place when the oil is in storage. H. M.

Production of Anhydrous Aluminium Chloride.—O. C. Ralston. *U.S. Bureau of Mines Technical Paper, No.* 321. 1923.

A study has been made of the uses to which aluminium chloride could be put provided the cost of production could be lowered sufficiently to make possible the commercial use of this material on the large scale. Production of low boiling from heavy hydrocarbons is chief amongst these. Descriptions of existing and proposed methods of manufacture are given. F. G. P. R.

Selenium Oxychloride for Cleaning Cylinders.—*Chem. and Met. Eng.*, 1923, **28**, No. 19, 849.

This solvent, discovered by Professor Lenher, of Wisconsin University, is excellent for removing rubber, resinous materials, glues, varnish, etc., and may also be advantageously used for freeing the cylinders of gas engines from carbon deposits. The latter usually consist of particles of carbon cemented together by hydrocarbons, such as partly carbonised oils or deposits from oils, including gasoline. Selenium oxychloride, alone or compounded with other substances, dissolves the binder constituents of the carbonised mass, which is thereby disintegrated and rendered easy to remove. F. G. P. R.

NAPHTHENIC ACIDS FROM JAPANESE PETROLEUM.—Y. Tanaka and S. Nagai. *J. Am. Chem. Soc.*, 1923, **45**, 754–6.

Alkaline waste lyes from the Akita petroleum refinery were acidified, the crude acids converted to their potassium salts in aqueous-alcoholic solution, from which neutral bodies were extracted by light petroleum. The acids were reprecipitated and distilled under reduced pressure in the presence of copper oxide. This process was repeated and the crude acids fractionated, that portion boiling at 170°–220° C./9 mm. being converted into methyl esters. By fractionation the following were obtained: methyl tri-, tetra- and pentadecanaphthenate, which are colourless liquids with a fruity odour. The pure acids are colourless and odourless, have no iodine number, and are not discoloured on exposure to air. F. G. P. R.

EFFECT OF FULLER'S EARTH ON PINENE AND OTHER TERPENES.—C. S. Venable. *J. Am. Chem. Soc.*, 1923, **45**, 728–738.

A study was made of the action of fuller's earth on α-pinene and other terpenes, both alone and in solution, at temperatures of –20°, –5°, 25° and 158° C. The reaction does not appear to go as imagined by Gurvich (*J. C. S.* abstracts, 1, 933), but as follows: (1) Intramolecular rearrangement to dipentene and terpene, with no formation of camphene. (2) Polymerisation to dipinene and other unidentified polyterpenes. (3) Slow decomposition of the polyterpenes at high temperatures, with formation of paraffin hydrocarbons and α-cymene, but not monoterpenes. The proportion of dipinene and polyterpenes to terpenes increases with rise in the temperature of reaction. Similar reactions occur with nopinene, dipentene, α-limonene, terpinene, camphene, sabinene, terpineol, borneol, and cineol, but α-cymene is entirely unaffected by fuller's earth. The maximum effect is obtained when fuller's earth contains 5–8% of combined water, which results by roasting the earth at 400° C. Diluents containing oxygen exert a strong inhibiting action. Iron and silica gels and activated charcoal do not react like fuller's earth. F. G. P. R.

THE GENESIS OF PETROLEUM.—E. Pyhälä. *Petroleum*, 1923, **19**, 495–499.

Mendelejeff, Byassons and Berthelot, supported by Sabatier, Eugen Coste and de Wilde, have advanced the theory of the inorganic origin of petroleum, by which it is supposed to have originated from the action of water at high temperatures on the carbides of manganese, samarium, uranium and other metals. The action of water on metallic carbides gives rise to the formation of acetylene, methane, ethylene, and solid and liquid hydrocarbons, free hydrogen and some resinous substances. A great objection to the inorganic theory of the origin of petroleum is the supposed fact that no oils of the type of lubricating oils and other hydrocarbons of high viscosity have been shown to originate by this process. The experiments of Ascham, Jpatiew and Routala show that many types of hydrocarbons may be obtained from acetylene, but not viscous oils of the type of lubricating oils. Acetylene at certain temperatures may be easily polymerised to benzene, toluol, etc., and at a temperature of 1000° C. condensed benzene nuclei, such as naphthalene may be formed. Sabatier and Senderens have shown that benzene hydrocarbons may be hydrogenised under certain conditions, and naphthenes may be formed in this way. Baku petroleum was subjected to cracking in the Nobel cracking plant at Baku. The aromatic hydrocarbons, benzene, toluene, and xylene were separated and subjected to a refining process, and afterwards distilled with steam and further refined. The

aqueous residue contained the light unsaturated hydrocarbons in a state of emulsion. This emulsion, being concentrated with superheated steam, gave 27·1 per cent. of distillate corresponding to solar oil and light oil, 21·1 per cent. of distillate corresponding to spindle oil, 12·3 per cent. corresponding to machine oil, and 14·1 per cent. corresponding to cylinder oil from the residue from crude benzol, and 47·4 per cent. light and solar oil, 16·8 per cent. spindle oil, and 8·2 per cent. machine oil from the residue from crude toluol. 10,250 kg. of mixed emulsion was distilled with superheated steam, and the oils obtained refined. 5·2 per cent. of a fraction analogous to cylinder oil was obtained, and 28·9 per cent. of a resinous residue. By these experiments it was shown that very mobile oils (1·0–1·2 Engler at 50° C.) might be polymerised to lubricating oils of high molecular weight. The residue of the light oil was redistilled after the xylene had been fractionated off, and other fractions up to 160° C., and a highly viscous drying oil was obtained. Other fractions yielded oxidised substances with the typical smell of naphthenic acids. From a mixture of the naphthenic acids of machine oil and refined kerosene a condensation product was obtained, possessing sp. gr. 0·8594 at 19·5° C., and an acid value of 0·27 per cent., soluble in concentrated sulphuric acid, insoluble in water and 96 per cent. alcohol. The distillation of a practically paraffin-free mazut gave machine oil with a solidification point of 0° C. It was found that the high solidification point was not caused by paraffin wax, but by ceresine with a m. pt. of 72·5° C., which had apparently been formed from asphaltic substances in the mazut during distillation. H. M.

THE PRODUCING SERIES OF THE APSHERON PENINSULA.—K. P. Kalitski. *P.S.J.*, 1922, T 111, p. 40.

The enormous amounts of petroleum produced around the city of Baku, in the Apsheron Peninsula, are drawn from a definite series of strata, known as the "Producing Series." The strata are of Pliocene age, and of a thickness exceeding 4,200 feet. They represent an alternation of clays and sands with intermediate layers of arenaceous clays and argillaceous sands. These strata are subject to great lateral variation; this, combined with the almost absolute absence of fossil evidence, or of characteristic intervening bands, which might serve to identify the producing strata in various areas throughout the peninsula, renders the study of this series a matter of extreme difficulty. The presence in the middle portion of the series of more coarse-grained sands with pebbles, makes it possible to subdivide it into three parts: the upper series, the "break" series (coarse-grained sands and pebbles), and the lower series. A number of other and more detailed divisions have been suggested, and an analysis and a comparison of these classifications will be found in the Russian text.

The question as to the kind of depositions represented by the producing series has also attracted the attention of investigators. Siogress classes them as littoral shallow water sediments, while Goubkin apparently considers them to be palaeogic deposits. Golubiatnikoff, on the other hand, suggests that they were formed at the bottom of pools and lakes, the "break" series being of Continental origin. Andrussoff ascribes the origin of the whole producing series to Continental deposition. Abramovitch is of opinion that the deposition of the series took place in a closed Continental basin. These contradictory views have induced the author entirely to revise the question of the origin of the producing strata. The insignificant horizontal extent of the series, combined with its remarkable thickness, its extreme similarity, lithologically speaking, to the strata

occurring in present-day deltas, as shown by borings (*e.g.*, in the deltas of the Po, the Ganges and the Nile) and the phenomena observed in our days of the Kura river and the Kizil-Agatch Bay, on the Caspian Sea, have suggested to the author the conclusion that the producing series of the Apsheron Peninsula belongs to deltaic and estuarine deposits. The whole of the producing series divides itself naturally into sediments formed in two deltas; the upper and the lower, which are superimposed upon each other. The lower delta is of limited extent horizontally, and inferior in point of thickness (200 sagens) to the upper delta, which extends westward far beyond the confines of the lower. The thickness of the upper deltaic deposits may be approximately estimated at 400 sagens. As to the so-called "break" series, the author considers it to be the base of the upper deltaic formation. In his opinion, the great thickness of the producing series is due to rapid sedimentation, and the resulting rapid increase of the load, which produced a subsidence of the sea-floor. The subsidence of the western coast of the Caspian Sea in the area under consideration is still continuing. It may be exemplified by the well-known subsidence of a caravansary in the Bay of Bibi-Eibat, and also by the submersion of the Baku Archipelago, then a great bulk of land with numerous mud-volcanoes, rise above the sea-level and form what is now called the Baku Archipelago. P. D.

"Sviatoi" Island.—N. M. Lodnioff. *P.S.J.*, 1922, T 111, p. 67.

"Sviatoi" island lies in the Caspian Sea, off the north-eastern coast of the Apsheron Peninsula, from which it is separated by the Apsheron Straits. The island extends in a north-westerly and south-easterly direction, which coincides with its main structural lines; *i.e.*, with the axes of two anticlinal folds proved in the central and north-eastern portions of the island.

The composing rocks are of Post-Pliocene and of Pliocene ages. The former is represented by sedimentation traceable to an ancient transgression of the Caspian Sea, and to a number of more recent invasions of the sea, which resulted in the deposition of sediments containing *Cardium edule*. The Pliocene is represented by what is called "the producing series," which is here divided into two sections. The upper beds are yellowish-grey, medium and cross-grained sands, with numerous inliers and concretionary bands of sandstone, the latter have been identified with those transitional depositions which, in the Apsheron Peninsula, separate the upper strata of the producing series (the Balakhani strata) from the lower beds (or the Kirmaku strata), the lower sands being a continuation of the Kirmaku strata, proved in the Apsheron Peninsula, are represented in the island by a frequent alternation of dark-grey sandy clays and fine-grained argillaceous sands. In the "Sviatoi" island, the lower strata (the Kirmaku strata) have been prospected by a number of wells drilled into the producing series through a thickness of 190 sagens; these wells have not yet reached the basal beds of the series. This figure (190 sagens) greatly exceeds the thickness of the Kirmaku series in the Apsheron Peninsula, where it ranges between 108 and 160 sagens.

As regards structure, we find in the "Sviatoi" island two ellipsoid anticlinal folds, one in the north, the other in the south, both elongated in a north-westerly and a south-easterly direction; they are not, however, in a line, but are displaced relatively to each other in a meridional direction, so that the southern extremity of the southern fold overlaps like the wings of a shifting scene. These folds are divided by a synclinal depression, which has a meridional direction, gradually merging into a south-

easterly one. The northern anticline occupies the north-eastern corner of the island, and its northern flank, the largest, runs out into the sea. The southern fold lies in the centre of the island and also extends under sea level, but changes its direction to a south-easterly one.

The main producing district (covering the northern anticline in the north-eastern corner of the island) has been subdivided by the writer into three distinct areas: the coastal area (plots Nos. 2 and 3), the central area (plots Nos. 1 and 4), and the southern area (plots Nos. 8, 7 and 6).

Most of the wells in the coastal area yield a low production, the output of a few ranges between medium and low, borehole No. 25 being the only one which has been steadily yielding a production between medium and good. The impregnation of the strata is apparently lowest in the coastal region.

In the central area all the wells give a moderately good yield, with occasional rises. The well yielding the highest production is No. 10.

The southern area comprises the best producing wells, with the exception of Nos. 12 and 22, which give but a poor yield. This is due to the fact that the sand tapped by these boreholes belong to the upper beds of the series and have been neglected by the other boreholes on account of their inadequate impregnation. As to the unsteady productions of wells Nos. 11 and 34, these must be put down to technical deficiencies. The author winds up by a detailed and circumstantial consideration of water-bearing sands. In his opinion, the greatest circumspection ought to be exercised in deepening the wells down to the lowest stratigraphical horizons, as the base of the series will soon have been reached, and the basal beds thereof may contain strong flows of water, which are a source of great danger, as the deposit may become waterlogged. P. D.

PROSPECTS OF GROZNI PETROLEUM INDUSTRY.—J. J. Yelin. *P.S.J.*, 1922, T 111, p. 114.

The Grozni petroleums, which may be divided into three principal varieties (oils of paraffin base, oils slightly saturated, and unsaturated oils), are at present subjected to a very elementary treatment, consisting in the separation of benzine and kerosene. The residue of paraffin oils, even if used as fuel, gives rise to considerable difficulties owing to its high freezing point.

A more efficient treatment of Grozni petroleums would greatly extend the possibilities of their use.

The benzine separated from petroleum contains a high percentage of light fractions, and may be made to yield motor fuel suitable for aviation and automobiles, and also petroleum ether; toluol may also be obtained therefrom. The residue of unsaturated petroleum may yield up to 30% solar oil, 6% spindle oil, and 18% machine oil. Oleaginous tar, the percentage of which reaches up to 50%, may be so handled as to give first-rate asphalt. Oils of paraffin base, if treated according to the new method of deparaffination which is now being tested by Prof. Gurvitch, should yield at least 2½% paraffin, not to speak of vaseline and asphalt, the deparaffinised residue being used as fuel. This process further permits the use of part of the half-products so obtained for the production of aromatic hydrocarbons, and the manufacture of artificial oil varnish.

All this points to the necessity of erecting, within the shortest possible time, extensive benzine and kerosene distilleries, as also lubricating oil, paraffin, and kindred works, while conducting careful laboratory investigations of the properties of Grozni petroleums. P. D.

THE COMPOSITION OF PETROLEUM.—J. S. Salkind. *P.S.J.*, 1922, T 111, p. 154.

The article opens by a systematic survey of all available evidence as to the nature of chemical compounds constituting various petroleums, followed by a detailed characterisation of the better-known varieties, such as Pennsylvania oils, Caspian oils and a few others. The author winds up by the following conclusions :—

(1) Oils of all varieties are mixtures of paraffin hydrocarbons, cyclic and polycyclic, of a chemically-saturated character, and of aromatic hydrocarbons.

(2) The percentage of saturated hydrocarbons in petroleums of all varieties is but low, not exceeding 20% even in the Pennsylvania oil, the richest in paraffin hydrocarbons.

(3) While exhibiting a great similarity of chemical composition, oils originating from different localities greatly differ as to the percentage of their constituents, a succession of transitional stages being observable between the highly-saturated Pennsylvania and the least-saturated California or Malay Archipelago oils.

(4) The absence of ethylene derivatives would seem to indicate that whatever the origin of petroleum it had been subjected to great chemical changes.

(5) While these changes were taking place, petroleum may have acquired absolutely novel constituents, such as sulphur compounds and, possibly, nitrogenous compounds.

(6) Such evidence as is now available as to the composition of crude petroleum does not warrant a preference in favour of either a mineral or an organic theory of its origin. P. D.

GAS DEPOSITS IN THE NORTHERN PORTION OF TAURIDA PROVINCE AND THE EXPLOITATION THEREOF.—A. J. Himmelfact. *P.S.J.*, 1922, T 111, p. 187.

While boring artesian wells in the Melitopol district (northern portion of Taurida Province), combustible gas was encountered in two areas lying south and south-east of the town of Melitopol, and covering in all 860 square kilometres. The gas occurs at a depth of 74 to 150 metres, and is chiefly composed of methane (up to 93/67%). Out of the 29 boreholes examined by the writer, the exploitation of gas was regularly conducted in two cases only ; in one instance it is used as motive power for a 45 h.p. gas-motor at a mill (this plant has been in operation for 15 years) ; in another, the gas is used for the lighting and heating of dwellings and offices at some salt workings. This gas may be utilised for two purposes ; either for central power plants, or for the condensation of gasoline. As all the wells were drilled for water, not for gas, none of the technical requirements which it is necessary to observe in boring for gas have been complied with, and it is imperative to drill a number of properly-equipped gas wells to ascertain whether this deposit is capable of yielding paying productions. At the same time, steps should be taken to develop the Crimean gas fields (Sivash, the Perekop isthmus, and Kertch) which, to all appearances, are geologically connected with the above deposit. P. D.

ON THE NATURE OF HYDROCARBONS PRODUCED BY THE DESTRUCTIVE DISTILLATION OF WEIMARN OIL SHALES.—S. P. Gwosdov and N. N. Nagozhov. *P.S.J.*, 1922, T 111, p. 194.

The present experimental investigation has been undertaken with a view to elucidating the nature of the hydrocarbons produced by destructive distillation of Weimarn oil shales. The material to be tested was

supplied by light fractions (below 170°) obtained in the course of destructive distillation at low temperatures and previously treated with weak acids and alkalis. The initial material consisting, as shown by preliminary tests, chiefly of unsaturated compounds, was broken up into fractions at intervals of 2°. The results of densometric, refractometric and cryoscopic tests to which individual fractions were subjected have forced upon the authors the conclusion that some of the essential constituents of shale-oil are hydrocarbons of the dihydro- and the tetrahydrobenzol series. P. D.

THE PETROLEUM HAND-DUG WELLS OF THE BAKU DISTRICT.—*Azerbaijan (Baku) Petroleum Industry*, 1923, No. 1, p. 53.

The cradle of the Baku petroleum industry was Surachani. Up to the annexation of Baku by Russia the petroleum hand-dug wells of the village Balachani, Sabuntchi, and others belonged to the Khan of Baku.

In 1842, 136 hand-dug wells, with a total production of 235,000 poods of petroleum, existed on the Apsheron Peninsula.

Up to 1872 the whole of the petroleum district was given as a private monopoly; but since 1872 large portions of the districts were divided into claims and sold by auction to various individuals. With the discovery of the application of boring tools in obtaining petroleum, and the rapid development of the boring systems in the Baku district, naturally the operations by hand-dug wells was practically discontinued. But with the growing value of petroleum in Baku during the last fifteen years the use of hand-dug wells has been considerably developed, with the result that on January 1st, 1917, there were 9,299 hand-dug wells in exploitation in the Baku district, and in addition, 580 hand-bored wells, the production of the same being as follows:—

	Poods.		Poods.
1909	1,000,000	1913	19,000,000
1910	3,000,000	1914	13,000,000
1911	6,000,000	1915	10,000,000
1912	15,000,000	1916	10,000,000

The number of people employed in the hand-dug wells industry was about 14,000. The depth of those hand-dug wells reached in some cases 350 feet, and the area occupied by them was about 1,254 acres.

The very large production of these shallow hand-dug wells shows the richness of these particular fields, and undoubtedly, in the near future this area will be developed by means of drilled wells. P. D.

"SVIATOI-OSTROFF" (HOLY ISLAND).

In a previous abstract the geological data of this island has been mentioned. The development of the petroleum industry on this island began in 1902.

By the end of last year there were 42 producer wells, having a depth ranging from 1,197 feet to 1,900 feet.

The total production since 1902 was 54,166,542 poods. According to Golubiatnikoff, based on past experience, by putting down 77 wells, a yearly production of not less 150,000,000 poods may be expected.

P. D.

HYDROCARBONS: METHOD AND APPARATUS FOR SELECTIVE OXIDATION OF.—W. A. Darrah. U.S.P. 1,448,655 of March 13, 1923. (Application Dec. 11, 1919).

Hydrocarbons are partially oxidised by introducing the substance into a rapidly moving current of preheated gases containing insufficient

oxygen for complete combustion. The pressure of the gases is lower at the point of introduction of the hydrocarbon than at any other point in the system. T. A. S.

GASES AND INFLAMMABLE LIQUIDS; IMPROVEMENTS IN STORAGE OF.—E. E. Petitpierre. E.P. 187,600 of Feb. 22, 1923. (Application Oct. 13, 1922; Convention Oct. 20, 1921.)

Peat, particularly yellow peat, is a satisfactory material for packing containers used for compressed gases or highly inflammable liquids. The peat, either in its natural condition or after suitable preparation by having been ground or compressed into lumps or reduced to peat wadding is washed, dried and mixed with other fibrous or porous materials. It is then rammed or compressed by hand into the container. After filling the receptacle with peat, the material is saturated with acetone or other inflammable solvent in which acetylene is dissolved. T. A. S.

OLEFINES; METHOD OF SEPARATING. L. C. Stewart, Assr. to Dow. Chemical Co. U.S.P. 1,452,322 of April 17, 1923. (Application Jan. 12, 1920.)

The olefine containing gas is passed through absorption towers in which it is brought in contact with cold petroleum oil capable of absorbing propylene and the higher members of the olefine series in preference to ethylene. T. A. S.

SUSPENSION AND EMULSION AND PROCESS OF MAKING SAME.—J. C. Morrell. U.S.P. 1,446,355–6 of Dec. 26, 1922. (Application (A) March 22, 1919; (B) June 22, 1920.)

(A) A dispersion of particles of hard pitch and oleaginous material is obtained in a medium containing a protective colloid, the medium forming the continuous or external phase.

A material for binding or agglomerating finely-divided substances is produced by preparing a suspension medium containing 1 to 5 per cent. of protective colloid, suspending in part of the medium 5 to 50 per cent. of finely-divided pitch, emulsifying in the remainder from 5 to 50 per cent. of oil. The suspension and emulsion are then mixed.

(B) Suspensions of non-fluid organic materials are prepared by adding such materials to a suspension medium containing a protective colloid, and grinding and agitating the mixture. T. A. S.

DISTILLATION AND REFINING.

HOW DO YOU OPERATE A COLUMN STILL?—F. H. Rhodes. *Chem. and Met. Eng.*, 1923, **28**, 590–592.

The partial condenser method is common practice in the operation of column stills. In this method the reflux which is supplied to the top of the fractionating column is condensed in a partial condenser or dephlegmator, from which the vapours pass on to a final condenser. This has the slight advantage of supplying a reflux of somewhat higher boiling point for fractionating, but in other respects the single condenser method is far preferable, as giving greater ease of control and constancy of the reflux-distillate ratio under varying conditions. With a ratio less than the optimum the purity of the product suffers, and a ratio in excess of the optimum, while but slightly increasing the purity, leads to a loss of heat and of time in the conduct of the distillation. In the single condenser method a dividing device may be installed below the condenser

to separate automatically the condensate into reflux and distillate, and may be set to maintain any desired ratio. The reflux ratio is varied to meet changes in the composition of the vapour entering the fractionating column, in the partial condenser method, by changing the rate of flow of the cooling water. The effect of this change is not immediate, and successful operation depends largely upon the judgment of the still-runner. The operation of the single condenser may be standardised and requires a minimum of attention, and the method may be advantageously applied to the distillation of high-boiling liquids. Naphthalene may be distilled by the single condenser method as easily as benzene, though its fractionation by the partial condenser method presents difficulty. With the object of recovering the heat contained in the vapour the single condenser may be built in two sections in series, the upper section being cooled by the ingoing charge to the still, and the lower section by water, or all the vapour may be condensed in a single condenser cooled by the ingoing charge, the condensed distillate only being passed through a water-cooler placed below the dividing device. The advantage of the higher boiling fraction being used as reflux in the partial condenser method may be compensated for in the single condenser method by adding one more plate to the column. H. M.

DISTILLATION EXPERIMENTS WITH THE HELP OF NATURAL GAS.—W. Friedmann. *Petroleum*, 1923, **19**, 367–375.

As the abundant natural gas of the Comodoro Rivadavia field does not find application in industry, attempts were made to utilise it in place of steam in oil distillation, and for the production of by-products from petroleum. The gas appears to have effected already a distillation of the light fractions from the crude oil, and the position of the primary oil deposits may, to some measure, be judged as the place at which the temperature corresponds to an equilibrium between the light and heavy hydrocarbons contained in the gas. When the gas issues from the well the hydrocarbons in it which are gaseous at 30°–35° C. carry with them heavier hydrocarbons out of the petroleum, which explains the fact that not only pentane may be condensed from the gas, but also hexane, heptane, octane, and nonane, with b. pts. from 125°–140° C., and even still higher homologues. To recover these various products from the gas the methods of compression, cooling and adsorption must be adopted, with regard to the critical temperatures of the constituents. The butanes may be liquified in the first and second stages of a compressor, while propane and ethane may be recovered in the third and fourth stages when cooled to 0° C. The gasolines containing such light constituents as propane and butane are very subject to "weathering," by which they may lose as much as 50%. Casing-head gasoline prepared by the cooling process, being free from these liquified gases, loses only 7%. Coconut charcoal has been used for the adsorption of the condensible constituents of natural gas. Fuller's earth exercises a selective adsorption of the lighter constituents of treated oil, and the same phenomenon is observed in the adsorption of gases by charcoal. Three absorbers are used, and the absorbed compounds successively driven off by superheated steam. A plant has been constructed to deal with 5 million cb. metres of gas per month, and to recover from this 500,000 litres of gasoline. Much greater quantities of gas are available, but the life of gas wells is uncertain. The content of gasoline per 1,000 cb. metres of gas varies from 33 to 95 litres. Apparatus for the experimental determination of the conditions of adsorption is described. A slow current of gas was found to increase the adsorption by 12%. Whereas

by ordinary distillation the Rivadavia oil only commences to yield its light constituents at 150°–160° C., by the new method 40% of the light fractions came off at 35°–40° C. The actual distillation of the oil commences at 90°–100° C. The water content of the oil may be distilled off at 40° C. by the current of natural gas, thus avoiding the difficulties from frothing later on. This method was successful with a strongly emulsified oil containing 30% of water, otherwise undistillable. Details are given of numerous experimental distillations. H. M.

Centrifugal Oil Treatment.—J. H. Shedgen. *Pet. Age* **11**, Nos. 2, 26–7, 78 (1923).

If crude oil is heated with acid in centrifugal mixers and subsequently heated with alkali in similar apparatus the resultant product gives, on distillation, fractions which do not require any further treatment.

T. A. S.

Improvements in the Benzene Recovery Process.—F. Raschig. *Gas u. Wasserfach*, 1922, **41**, 655–658.

In the benzene recovery process from coke-oven gas, as usually conducted, the scrubbing oil only attains a concentration of 2% of benzene, and the amount of steam used in distilling the benzene off amounts to five times the weight of benzene, while the water to cool the scrubbing oil amounts to some 100 times the amount of benzene. Theoretically the steam required should be 20%, and the cooling water 120% only of the benzene. The author proposes to use scrubbers much smaller than the customary towers 10 metres high, to fill them with Raschig's rings, and to circulate the scrubbing oil briskly through them, pumping it from the bottom to the top of each scrubber, three scrubbers being arranged in series, and the scrubbing oil and gas passing through them in countercurrent. The scrubbers are about 2 m. in diam., and 4 m. high. They have a perforated division at 1 m. height, and on this rests a mass of Raschig's rings, of sheet iron or porcelain, 3 m. high. The scrubber has a conical top, in which is a sprinkler from which the oil is sprayed, and the gas enters at the bottom and leaves the top to enter the bottom of the next scrubber in series. This apparatus is capable of dealing with 100,000 cb. m. of gas per 24 hours, and 24 cb. m. of oil is pumped through the plant each hour. 4 cb. m. of oil is withdrawn each hour for benzene recovery, and a concentration of 3% benzene is reached. The apparatus, being smaller, is cheaper than the ordinary plant. In the recovery apparatus the oil runs continuously into a vacuum pan indirectly heated by steam. A large heat interchanger serves to transfer heat from the spent oil to the oil entering the recovery plant, and the benzene vapours, owing to the indirect steam heating, are practically free from water vapour. 3,000 kg. of benzene is recovered per 24 hours. The last traces of benzene are removed in a worm condenser. A portion of the naphthalene is separated from the oil by cooling, and the remainder, dissolved in the carbolic oil, is withdrawn for carbolic acid manufacture and naphthalene recovery. The expenditure of steam is 1¼ times the recovered benzene, and the cooling water employed is 10 times the latter amount. H. M.

Treatment of Natural-Gas Gasoline to meet "Doctor" Test.—D. B. Dow. *Nat. Pet. News*, 1923, **15**, No. 20, 99–111.

Owing to the rapidity with which gasoline produced from natural gas goes off colour and becomes sour in smell, as well as "sour" to the "doctor" test, it was considered by the author that a full description of most of the known methods of purifying this gasoline in order to render it stable during storage would be of great use, especially to the small

producer. The paper contains sections on methods of testing for sulphur, the removal of sulphur from gasoline, situation and design of the treating plant, preparation of the reagents, operation of the plant, and finally the total cost of operating. The major portion of the paper deals with the use of "doctor" solution—*i.e.*, a caustic soda solution of litharge containing sulphur, as the reagent for sweetening gasoline, and the original paper should be consulted for full details of the method of treatment.

F. G. P. R.

DESULPHURISING PETROLEUM PRODUCTS.—H. Broche. *Brennstoff und Chemie*, 1923.

Experiments were carried out on an asphaltic Mexican crude in which the sulphur content was not appreciably lowered by the Frash method of treating with sulphuric acid, followed by washing with sodium plumbite with the addition of flowers of sulphur. The oil was separated into fractions which contained from 0·75 to 3·31 per cent. of sulphur. Treatment with 4 per cent. by volume of sulphuric acid, followed by caustic soda, reduced the sulphur in one case from 1·69 to 0·22 per cent., and in another from 2·2 to 0·73 per cent. By the Frash process the reduction was only from 1·6 to 1·4 per cent., but subsequent distillation gave a distillate with 0·35 per cent. of sulphur. Distillation alone of untreated oil hardly altered the sulphur content, thus proving that the chemical treatment had radically changed the constitution of the sulphur compounds. After acid treatment an oil was washed in separate portions with (1) caustic soda alone, (2) sodium plumbite and flowers of sulphur, and (3) sodium plumbite alone. Each portion was then distilled, and the distillates were found to contain (1) 0·37 per cent. of sulphur, (2) 0·68 per cent., and (3) 0·37 per cent. Hence the use of sodium plumbite appears to have no advantage over caustic soda, whilst the addition of flowers of sulphur is actually harmful to the process.

F. G. P. R.

FRACTIONAL CONDENSATION OF PETROLEUM OR OTHER VAPOURS—IMPROVED APPARATUS FOR.—THE GRISCOM-RUSSEL CO. Assees of C. Jefferson. E.P. 174,569 of March 12, 1923. (Convention (U.S.) Jan. 25, 1921.)

The apparatus consists of a dephlegmator column containing a large number of vertical tubes. The column is divided into two portions by a diaphragm, the upper portion being cooled. The cooling medium is caused to take a tortuous path by means of a helical baffle through which the tubes pass. For convenience of construction the helical baffle is built of sections, each consisting of a single turn. These sections may be stamped out of sheet steel in one operation. Expansion and contraction of the tubes is allowed for by having a movable head, into which the tubes are welded or expanded. Escape of vapours at the movable head is prevented by means of a packing ring. Vapours from the still enter the side of the bottom section of the dephlegmator and pass upwards through the tubes. Uncondensed vapours are removed from the movable head. By varying the amount of coding medium any desired portion of the vapours may be condensed. The condensed liquid returns to the bottom section of the dephlegmator at the base of which is a draw-off pipe.

T. A. S.

DESULPHURISING HYDROCARBONS AND CRUDE PETROLEUM.—P. von Ditmar. U.S.P. 1,448,643 of March 13, 1923. (Application Feb. 6, 1923.)

The sulphur-containing hydrocarbons are heated in closed vessel, with metallic soaps of the higher fatty acids. The fatty acids liberated are drawn off when a layer has formed. During the reaction a small quantity of metallic sodium is added.

T. A. S.

DESULPHURISING OIL—IMPROVEMENTS RELATING TO.—N. H. Freeman. E.P. 193,979 of March 5, 1923. (Application Dec. 5, 1921.)

The oil to be desulphurised is atomised through fine jets, under a pressure of about 50 lbs., into a desulphurising medium. Gaseous liquid and solid desulphurising media may be used. When gaseous media, such as ammonia, are used, water is also sprayed in. Free hydrogen also facilitates the removal of sulphur. The operation is carried out at atmospheric temperatures and the desulphurising medium is retained at atmospheric pressure. T. A. S.

REMOVAL OF PARAFFIN FROM MINERAL OILS.—M. A. Rakusin. E.P. 193,896 of March 5, 1923. (Application Oct. 3, 1921.)

The flow-point of paraffin oils is raised by the removal of paraffin wax and bitumen. Crude oil, from which the fractions boiling below 160° C. have been removed, is diluted with alcohol, turpentine, or a mixture of both. The paraffin wax and bitumen are precipitated, and can be separated by filtration or centrifugal force. The alcohol and turpentine are recovered by steam distillation. The separated wax and bitumen are distilled *in vacuo*, hard paraffin, which can be easily refined, distilling over and a high melting neutral bitumen being obtained as a residue. T. A. S.

TRANSPORTING AND STORING PETROLEUM OIL—PREVENTION OF LOSSES.—F. M. Seibert and E. T. Gregg. U.S.P. 1,439,451 of Dec. 19, 1923.

The loss of volatile liquid hydrocarbons from petroleum oils during storage or transportation is prevented by passing the vapours given off through chambers containing absorbent material such as activated charcoal or silica gel. T. A. S.

PETROLEUM OIL—PROCESS FOR REFINING.—J. C. Black. U.S.P. 1,447,118 of Feb. 27, 1923. (Application June 20, 1922.)

Fractional condensation of the vapours obtained on distilling oil is brought about by bringing the vapours in contact with liquid hydrocarbon of approximately the same composition as the fraction it is desired to separate. T. A. S.

RESOLUTION OF WATER-IN-OIL EMULSIONS.—H. A. Gill. Comm. by The Sharples Speciality Co. E.P. 195,876 of April 12, 1923. (Application April 25, 1922.)

The stability of emulsions is dependent on the properties of the colloid emulsifying agent present. The effect of a colloid emulsifying agent present in the oil, and giving an emulsion in which the oil is continuous, the water dispersed, may be counteracted by the addition of a colloid soluble in the water and tending to make the water take the continuous condition. Such colloids are glue, albumen, gelatine, casein and gums. The addition of one-tenth of one per cent. of glue to a water in oil emulsion renders it possible to separate the emulsion either by gravity or centrifugal force. The addition of such substances as caustic soda to emulsions obtained in the acid treatment of oil is thus avoided. T. A. S.

OIL REFINING DEVICE.—C. F. Hillman. U.S.P. 1,451,574 of April 10, 1923. (Application March 13, 1920.)

Oil, preheated by means of a still residue, and also by means of still vapours, is passed through a pipe still and thence through a separator. The vapours from the separator are first used for preheating purposes,

and then passed through a second separator to a condenser. Oil collecting in the second separator is returned to ordinary stills for distillation. The oil collecting in the first separator is not redistilled. T. A. S.

Oil Distillation—Apparatus for.—J. W. Van Dyke. Assr. to The Atlantic Refining Co. U.S.P. 1,451,129 of April 10, 1923. (Application August 15, 1919.)

Oil is raised to the temperature of distillation, and steam at a lower temperature admitted. The vapours are partially condensed so that oil separates and the uncondensed steam is drawn into a current of moving steam and returned to the still. T. A. S.

Refining of Crude Oil—Apparatus for.—I. Mayes. U.S.P. 1,451,739 of April 17, 1923. (Application Sept. 13, 1920.)

Oil is passed through a series of shallow stills supported over furnaces. The bottoms of the stills are fitted with U-shaped members to cause the oil to take a tortuous path through the still. T. A. S.

Dehydrator.—Assr. to Petroleum Rectifying Co. U.S.P. 1,452,207 of April 17, 1923. (Application Oct. 28, 1920.)

Oils are dehydrated in a chamber fitted with a propellor and a rotating electrode. T. A. S.

Centrifugal Separation of Wax from Oil.—C. A. Porter and O. C. Brewster. Assrs. to P. Sullivan and G. R. Hagens. U.S.P. 1,452,388 of April 17, 1923. (Application Nov. 19, 1921.)

Oil containing wax is diluted with a solvent to produce a liquid of low viscosity. It is then cooled until the greater part of the wax separates and passes through a centrifugal separator while causing the mixture to float on a non-progressing layer of aqueous fluid. The clear oil and solvent is withdrawn from the centrifugal and the waxy material is passed to a separate compartment where sufficient warm water is passed in to melt the wax when it is expelled by centrifugal force. T. A. S.

Improved Production of Volatile Liquid Fuels from Mineral and Other Oils.—F. M. Accioly. E.P. 193,934 of Feb. 26, 1923. (Application Nov. 26, 1921.)

Potassium carbonate, sulphuric acid, and naphthalene are added to crude oil, *e.g.*, shale-oil, vegetable or animal oil, or mixtures, and product submitted to distillation. Several recipes are given for the treatment of 100 gals. of oil, naphtha of sp. gr. 0·800 to the extent of 25 to 40 gals. being added. A product which is not gasoline, but having the same combustible power and sp. gr. 0·735–0·800, is obtained, and exceeds the natural gasoline content of the oil by 1 to 4 times. The residues are worked up to yield kerosene, lubricating oil, grease, paraffin wax, ichthyol and tar. T. A. S.

Desulphurising Mineral Oils and Spirits.—A. C. Nesfield. E.P. 196,680 of April 25, 1923. (Application Jan. 25, 1922.)

Mineral oils and spirits, more particularly those derived from shale, are desulphurised by treatment with hydrochloric acid mixed with air or hydrogen and air and subsequently washing the oil with caustic soda or milk of lime, and filtering to remove any solid matter. The treatment is carried out in a vessel fitted with screens to ensure the intimate mixing of the oil and gas. The gas is admitted at the bottom of the vessel, and

a slight vacuum maintained to ensure its removal. The proportion of hydrochloric acid gas to air varies from 1 to 10 to 1 to 100, according to the nature of the oil to be treated, the necessary concentration having to be determined by experiment. When water-white spirits are desired the above treatment is followed by a treatment with 2% of sulphuric acid. Any emulsions formed in the process can be broken by washing with salt solution. T. A. S.

TREATMENT OF HYDROCARBON OILS.—J. P. Persch. U.S.P. 1,454,485 of May 8, 1923. (Application Feb. 10, 1921.)

The passage of heavy oils through long horizontal pipes is facilitated by the admission of hot gas. T. A. S.

PURIFICATION OF MINERAL OILS.—E. E. Arnold. Assr. to The Nitrogen Corporation. U.S.P. 1,454,593 of May 8, 1923. (Application Feb. 24, 1921.)

Oil is treated with liquid anhydrous ammonia in the presence of an alkali-metal oxygen bearing compound. Under the oxidising conditions the sulphur in the oil unites with the ammonia to form a compound. T. A. S.

RESOLUTION AND PREVENTION OF THE FORMATION OF EMULSIONS.—E. E. Ayres. Assr. to The Sharples Specialty Co. U.S.P. 1,454,616–7 of May 8, 1923. (Application Feb. 10, 1921 and Feb. 24, 1921.)

Sodium resinate is dissolved in a substance adapted to produce a solution that is miscible with petroleum. On adding this solution to an emulsion the constituents can be separated by subsidence. The addition of the solution to petroleum prevents the formation of water-in-oil emulsions. T. A. S.

ELECTRIC DEHYDRATOR.—F. W. Harris, Assr. to Petroleum Rectifying Co. U.S.P. 1,455,139 of May 15, 1923. (Application October 19, 1920.)

The dehydrator consists of an inner and outer electrode, the inner electrode being supported so that an electric field of uniform thickness is formed between the electrodes. The emulsion passing between the electrodes is agitated. T. A. S.

EXTRACTION OF VAPOURS FROM GASES—PORTABLE APPARATUS FOR.—G. G. Oberfell and G. A. Burrell, Assrs. to Gasoline Recovery Corporation. U.S.P. 1,455,407 of May 15, 1923. (Application Aug. 2, 1920.)

An absorber unit containing activated charcoal is mounted on a portable platform. A boiler on the platform supplies steam for distilling off the gasoline absorbed on the charcoal. T. A. S.

FILTRATION.—G. H. Fifield, Assr. to Standard Oil Co. U.S.P. 1,455,436 of May 15, 1923. (Application Nov. 5, 1920.)

Chilled oil is forced through a filtering surface, wax being deposited. A heat conductor extending through the filtering surface is used in removing the wax by melting it. T. A. S.

BITUMEN AND CELLULOSE CONTAINING SUBSTANCES—PROCESS OF TREATMENT OF.—J. Moscicki. U.S.P. 1,455,546 of May 15, 1923. (Application Aug. 25, 1921.)

The vapours from the distillation of bitumen and cellulose containing substances are separated into their component parts by passing them upwardly through dephlegmator columns. Condensate collected at the

bottom of the column is cooled to slightly below its condensation temperature and then pumped to the top of the column, where it meets the rising vapours. T. A. S.

OIL—CONTAINING WATER; METHOD OF TREATING.—O. C. Gatrell. U.S.P. 1,440,352 of Dec. 26, 1922. (Application Nov. 1, 1920.)

Oil is dehydrated by heating and agitating it, powdered Portland cement and slaked lime being added. T. A. S.

CRACKING.

CRACKING OF PETROLEUM.—C. Kroll. U.S.P. 1,448,128 of March 13, 1923. (Application Feb. 4, 1919.)

The oil to be cracked is maintained under pressure in a large, approximately horizontal, insulated reaction chamber. The chamber is traversed by a horizontal flue through which hot gases are passed; a rotating scraper is provided for removing carbonaceous matter from the walls of the flue to a relatively inactive portion of the reaction chamber. T. A. S.

LOW-BOILING HYDROCARBONS—PRODUCTION OF FROM HIGHER BOILING HYDROCARBONS.—M. Melamid. E.P. 193,922 of March 8, 1923. (Application Nov. 10, 1921.)

In previous patents the hydrogenation of tar, mineral, and other oils in the presence of tin or alloys is described. The present patent protects the cracking of hydrocarbons at high temperatures without pressure and in the presence of tin. The temperature used must be above 600° C. and 800° C. is preferable. At 900° C. the use of hydrogen is unnecessary. T. A. S.

LOW-BOILING HYDROCARBONS—METHOD OF PRODUCTION OF—FROM HIGH-BOILING HYDROCARBONS.—M. Melamid. E.P. 180,625 of Feb. 12, 1923. (Application Nov. 10, 1921; Convention May 25, 1921.)

High-boiling mineral oils or tar oils are heated to suitably high temperatures in the presence of metals or alloys, which melt below 700° C. Zinc and lead are unsuitable as they form carbides, but soft solders and alloys of bismuth may be used. T. A. S.

STILL FOR HYDROCARBONS.—H. B. Setzler. Assr. to the National Refining Co. U.S.P. 1,449,452 of March 27, 1923. (Application Jan. 14, 1918.)

Heat is conserved in a pressure still by causing the vapours from the still to preheat the oil entering the still. This effect is obtained by fitting the still with a still head consisting of two vertical columns, the vapours entering the bottom of one column and pass from the top of this column to the top of the other. From the bottom of the second column the vapours pass a condenser of the ordinary type. The oil to be preheated is pumped through coils in the columns in a direction countercurrent to the vapours. T. A. S.

APPARATUS FOR REFINING HYDROCARBON OILS.—V. F. Clemons. Assr. to C. F. O'Neall. U.S.P. 1,449,734 of March 27, 1923. (Application May 22, 1922.)

Crude oil or hydrocarbon oils are distilled in a pressure still, in the vapour space of which a bed of fuller's earth is placed supported on a

foraminous stage. The vapours, after passing through the fuller's earth, escape by a valve, which opens at a predetermined pressure, to a condenser. T. A. S.

CRACKING HYDROCARBON OILS UNDER PRESSURE.—R. W. Hanna, W. D. Mason, and W. G. Hamilton. Assrs. to Standard Oil Co., San Francisco. U.S.P. 1,449,226–7 of March 20, 1923. (Application Aug. 29, 1921.)

Hydrocarbon oils are cracked by a continuous process under pressure. A moving stream of the oil is heated in a tube to a temperature above the boiling-point of some of the fractions, but a sufficient pressure is maintained to keep the liquid substantially in the liquid phase. The heated oil is then passed to a cracking system, which includes a heating zone, a vapourising chamber, and a condenser maintained under pressure. The oil in the heating zone is heated to cracking temperature, over-heating and consequent deposition of carbon being avoided. On leaving the cracking zone the oil enters the vapourising chamber.

In patent No. 1,449,227 the unevaporated portion of oil from the evaporating chamber is pumped back through the heating zone, the process thus becoming cyclic. Cracking takes place substantially in the evaporating chamber, the oil being pumped at such a rate that it is only in the heating zone for a period of ten minutes. The heating is so controlled that the oil leaving the heating zone is not more than 15° F. higher in temperature than on entering the heating zone. T. A. S.

CRACKING HYDROCARBON OILS, COAL-TAR, ETC.—W. F. Faragher and W. A. Gruse. E.P. 174,086 of March 22, 1923. (Convention (U.S.) Jan. 15. 1923.)

The deposition of carbon during cracking is prevented by maintaining a circulation of the oil by means of an air lift. The operation is carried out in a boiler of the water tube type, the 'air lift' effect being obtained by the introduction of jets of gas or by the internal generation of gas. The gas also tends to carry away the low-boiling fractions as they are produced. T. A. S.

PRODUCTION OF LOW-BOILING HYDROCARBONS.—M. Melamid. E.P. 174,321 of March 8, 1923. (Convention (Germany) Jan. 15, 1921.)

Processes of cracking in the presence of tin or alloys are described in patents Nos. 171,367 and 171,390. In the present patent cracking is carried out at atmospheric pressures, in the presence of a large excess of hydrogen and tin at a red heat. Intimate contact between the vapour and the metal is obtained by making the vapour traverse a tortuous path over superposed trays of the metal placed one centimetre apart. T. A. S.

PETROL—IMPROVED PRODUCTION OF.—F. Tinker. E.P. 195,465 of April 5, 1923. (Application Jan. 7, 1922.)

Crude mineral oil or the like is passed through a pipe still into a separating chamber from which vapours pass to a fractionating column. Liquid condensed in the column is returned to another still, the vapours from this still passing through a superheater and thence to either the fractionating column or the separating chamber. Residue from the separating chamber is used in a heat exchanger to preheat the incoming oil. The ordinary type of crude oil still may be used instead of a pipe still. A higher yield of petrol is obtained owing to the cracking which takes place in the superheater. As a rapid flow of liquid through the second still and superheater may be maintained, carbonisation can be reduced to a minimum. T. A. S.

HYDROCARBON OILS—PROCESSES AND APPARATUS FOR CONVERSION OF.—V. L. Emerson. E.P. 163,277 of Feb. 8, 1922. (Application Nov. 8, 1920; Convention May 6, 1920), and E.P. 168,573 of March 22, 1922. (Application Dec. 22, 1920; Convention May 6, 1920). See U.S.P. 1,367,806–7.

Oil is heated in a pressure still, and the evolved vapours are expanded into an expansion chamber containing oil to be preheated before entering the pressure still. The expansion takes place through ejector-nozzles, the oil to be heated being picked up and blown through the nozzle with the gas stream. Vapours from the expansion chamber containing the cracked products and volatile portions and impurities of the incoming oil are taken to a condenser. The preheated oil is pumped to the pressure still. In 168,573 a modified form of expansion chamber is used.

In both forms of apparatus opposed pairs of jets are used, the impact of the gases after expansion assisting in the cracking.

When the cracking still is operating on fuel oil a pressure of 75 to 100 lbs. is used, and a temperature of 700–800° F. The velocity of the gases at the jets is approximately 2900 feet per second. T. A. S.

IMPROVED PROCESS FOR CONVERTING HEAVY HYDROCARBON OILS INTO LIGHTER HYDROCARBON OILS.—Standard Oil Co. E.P. 174,589 of April 16, 1923. (Convention Jan. 15, 1921.)

Oil at a temperature of less than 700° F. is fed into a conduit where the temperature is raised to 750–900° F. The passage through the conduit is so regulated that not more than 10% of the oil is converted into gasoline whilst in the conduit and the substantial deposition of carbon is prevented. After leaving the conduit the oil enters a chamber the walls of which are maintained at a temperature of 900° F. Further cracking takes place, the yield of gasoline being doubled. Pressure is exerted to maintain the unchanged charging stock in the liquid phase and the converted products are continuously drawn from the reacting chamber. T. A. S.

LOW-BOILING HYDROCARBONS—PRODUCTION OF—FROM HIGH-BOILING HYDROCARBONS.—Plausons (Parent Co.). E.P. 193,071 of Feb. 16, 1923. (Application Aug. 16, 1921.)

Heavy oils, *e.g.*, tar, shale or mineral oils, are heated with a hygroscopic mixture containing a metallic oxide and chloride. A mixture of zinc chloride and lime appears to be best for the process. The result can be achieved by the use of either material alone, but a mixture gives better results. The oil should be dehydrated before treatment, and if much phenol is present the vessel should be tinned. A Bavarian shale oil (b.p. 130–350° C.) when treated with 10 pts. $ZnCl_2$, 5 pts. $MgCl_2$ and 40 pts. quicklime give the following fractions:—

B.P. 60° —140° C.	58%
B.P. 140° —260° C.	42%

T. A. S.

CRACKING HYDROCARBONS.—H. A. Weir and S. A. Weir. U.S.P. 1,454,142 of May 8, 1923. (Application April 12, 1920.)

The apparatus consists of a large horizontal still, above which a smaller horizontal still is mounted. Vapours from the larger still are delivered into the smaller still by pipes which terminate above the bottom of the secondary still. The furnace gases proceed under the bottom of the larger still, and are brought back along the outer walls of the smaller still to a stack. The ends of the smaller still project through the still-

housing so that they are accessible for cleaning. Vapours from the secondary still are taken through a dephlegmator to a condenser. The apparatus works under pressure. When a certain amount of cracking has been brought about the apparatus is used as an ordinary still for the recovery of kerosene, solar oil, etc. from the residues. T. A. S.

METHOD AND APPARATUS FOR PRODUCING A CHEMICAL UNION BETWEEN HYDROCARBON GASES AND HYDROCARBON OILS.—H. B. Snyder. U.S.P. 1,454,567 of May 8, 1923. (Application Dec. 27, 1921.)

Hydrocarbon gas, *e.g.*, methane, is subjected to a high pressure and a quantity of hydrocarbon oil introduced. An electric arc is passed through the gas-oil mixture whilst under pressure. T. A. S.

ELECTRICALLY-OPERATING TREATING CHAMBER FOR HYDROCARBON VAPOURS AND GASES.—J. L. McCabe. U.S.P. 1,455,088 of May 15, 1923.

A tubular treating chamber, having walls of non-conducting material, is fitted with an axially-arranged insulating element, which is surrounded by a space. A heating coil is wound on the insulated element. This coil also serves as an electrode of another circuit, the other electrode of which is wound on the outside of the treating chamber. T. A. S.

PROCESS AND APPARATUS FOR CONVERTING OILS.—J. H. Adams, Assr. to The Texas Co. U.S.P. 1,455,376–7 of May 15, 1923. (Application Dec. 29, 1917.)

Preheated oil is sprayed, either with or without superheated steam, on to a heating element heated to cracking temperature. Vapours from the heating element rise to a dome where condensation of the uncracked portion takes place, the condensate flowing back into the vicinity of the heating element. Cracked vapours pass to a condenser. The whole of the apparatus is maintained at pressures higher than atmospheric. T. A. S.

MOTOR FUELS.

MOTOR SPIRIT—IMPROVEMENTS RELATING TO.—A. G. Burnell and W. Dave. E.P. 193,955 of March 1, 1923. (Application Dec. 1, 1921.)

The process is an extension of Pat. No. 171,566 for the conversion of the liquid condensed, under a pressure 150 lb. per sq. in., from oil gas. This liquid is unsuitable for use as motor fuel owing to the gumming residue left on evaporation. These constituents are removed by treating the liquid with anhydrous metallic chlorides—*e.g.*, aluminium chloride with or without the aid of chlorine gas. The process is preferably carried out at atmospheric pressure, but higher pressures may be used. The oil is refluxed with the aluminium chloride in a still, chlorine being passed during about one-quarter of the refluxing time, and preferably in the middle of the refluxing period. The aluminium chloride, in the proportion 2 ozs. to the gallon, is mixed with petrol and added as a thin cream. During the refluxing the contents of the still are agitated with a mechanical stirrer. After the operation the product is distilled, the fraction boiling between 70–140° C. being collected. Satisfactory treatment is determined by testing the distillate for bromine absorption and the formation of gumming residues on evaporation. T. A. S.

LIQUID FUEL.—M. L. Valdes and V. B. Villar. U.S.P. 1,448,245 of March 13, 1923. (Application Nov. 14, 1921.)

The fuel consists of nitro-benzene, ethyl alcohol, and nitrous ether. T. A. S.

Lubrication.

Comparative Examination of Mineral and Vegetable Lubricating Oils. M. A. Rakusin. *Petroleum*, 1923, **19**, 454–456.

Owing to the scarcity and high prices of vegetable oils used for lubrication, such as castor oil, with a flash point of 270°–280° C. and a minimum coefficient of friction, experiments were made in the refining of mineral oils with a view to their substitution for the more expensive products. A cylinder oil was found to possess a much flatter viscosity curve after thorough refining than before refining, the viscosity before refining being 16·82 min. at 80° C. and 1·67 min. at 190° C., and after refining 8·46 min. at 80° C. and 1·36 min. at 190° C. The loss in refining is amply compensated for by the increased value of the resultant oil. Of the vegetable oils tested castor-oil gave the flattest viscosity curve. For the lubrication or cooling of working metal surfaces the superiority of rape oil over mineral oil is undoubted. Blown rape oil has a lower flash point and higher viscosities at all temperatures than the untreated product, and so cannot be employed where much heat is generated. When blown oils are used for lubrication the metal surfaces may get covered with a film of varnish, and where blown oils are employed to increase the viscosity of mineral oils they are liable to separate from the mixture. H. M.

Testing the Quality of Lubricating Oils.—W. H. Herschel. *Proc. Eng. Soc. W. Pa.*, 1923, **38**, 503–518.

Liquid lubrication falls into two classes: complete film lubrication at high speeds or low pressures, and incomplete film lubrication due to low speeds, high pressures, or inadequate supply of lubricant. The lubricant must be suited to its purpose and must retain its original properties for a sufficiently long period. It must be fluid enough to reach the rubbing surfaces, and the lower the coefficient of friction the better. Gravity has been discarded as a basis for fuel-oil specifications, as there is no relation between gravity and viscosity, but gravity is an indication of the source of the crude oil from which a lubricant is derived. The modulus of the Baumé scale has been settled, the scale based on a modulus of 141·5 being known as the A.P.I. scale, and that with a modulus of 140 as the Baumé scale. The gravity is a factor in the determination of absolute viscosity from viscometer readings. Grades of colour are best estimated by the use of standard potassium dichromate solutions, but the importance of colour is not great. As the solutions change colour on standing, some oil must be used as a secondary standard for comparison. The flash point is a measure of the volatility of the most volatile constituent, and the fire point serves as a check on the flash point. Naphthene base oils are, as a rule, lower in fire and flash points than corresponding paraffin base oils. The pour point, or temperature of solidification, tends to be accepted in place of the melting point. It is important as a measure of the suitability of the oil for winter use out of doors, but a very viscous oil at a temperature above its pour point may offer more resistance to motion than a less viscous oil below its pour point. A very low pour point can only be obtained by the complete removal of paraffin wax. Viscosity is not a quality test unless it be determined at two temperatures. The addition of fatty oils in compounding decreases the change of viscosity with the temperature. For a given machine there is a viscosity of minimum friction, and the ratio of the viscosity of the oil film to this may be regarded as the factor of safety. Five has been suggested as a suitable factor of safety. Viscometers tend to show too high a time of flow after prolonged use, owing to adsorbed films.

A table is given for the correction of Saybolt viscosity to normal. The property of oiliness has no effect during complete film lubrication, but there is liable to be incomplete film lubrication on all machinery on starting, and sometimes during operation. Oiliness appears to be due to adhesion, adsorption, or a segregation of some constituent of the lubricant at the boundaries. Emulsion tests are intended to indicate the readiness with which the oil will separate from water, as for turbine oils, and may be carried out by agitation by steam or mechanical agitation, and with water, or a solution of caustic soda. In the latter case a soap is formed from the free naphthenic acids present. The emulsion test is a sensitive indication of organic acidity. Oils of high organic acidity are not durable in service and tend to form sludge. Sludging is generally the result of the formation of oxidation products of an asphaltic nature. Acidity is an indication of chemical instability. The carbon residue test is a cracking test. So-called carbon deposits are composed largely of asphaltic oxidation products. The Waters carbonisation test is for the determination of asphaltic matter insoluble in petroleum ether, and determines chemical instability. There is no correlation between carbonisation and demulsibility. The Waters test appears to be the best yet proposed to measure the resistance of an oil to oxidation.

H. M.

Comparison of Paraffin and Asphalt Base Lubricating Oils.—F. N. Williams. *Refiner*. 1922. 1. No. 3. 13.

The two oils under comparison had the same viscosity at 100° F., 361 Saybolt seconds. The change in viscosity between 70° F. and 100° F. was 1427 secs. for the asphalt base oil and 620 secs. for the paraffin, whilst between 100° F. and 212° F. the changes were 312° F. and 309° F. respectively. Therefore, over the normal working range of temperature there is very little difference between the two oils. Where a low "cold test" oil is required the asphalt base was superior, having a pour point of 20° F. against 40° F. of the paraffin. In the Conradson test the asphalt also gave a better result, 0·063 per cent. of soft and flaky carbon against 0·130 per cent. of hard and gritty carbon, of which the former would undoubtedly cause less damage to cylinder walls and be easier to remove.

F. G. P. R.

Friction Testing of Lubricating Oils.—W. H. Herschel. *Chem. and Met. Eng.*, 1923, **28**, 594–598.

For the estimation of the property of "oiliness," an oil friction-testing machine of the Cornell type was used. The journal had a diameter of 3·748 in., and was 3·48 in. in length. The width of bronze bearing block was 2·01 in., and net projected area 6·1 sq. in. The maximum load was 715 lb. per sq. in., and the speed varied from 43–814 ft. per min. The arc of contact was 65°. The most interesting results being gained near the point of seizure, a bearing block of Ulco metal was used to guard against roughening of the journal. The coefficient of friction was found to be about 0·0007 less on decreasing than on increasing loads, and tests made with decreasing load were corrected by this amount. The coefficient of friction was calculated and plotted against

$$S = \frac{\mu n}{p}\left(\frac{d}{2\Delta}\right)$$

where μ = viscosity in poises, n = speed in revs. per second, p = pressure in dynes per s.q cm., and $\frac{d}{2\Delta}$ = ratio of diameter of journal to difference

between diameters of bearing and journal. Tests were made on a mineral oil of paraffin base, one of naphthene base, and cotton-seed oil, all three being diluted with 300° mineral seal oil, or kerosene and gasoline, to vary the viscosity. The minimum coefficient of friction was 0·00094, at a value of S = 0·00173 for a mixture of petroleum oil and kerosene of the same viscosity as straight 300° oil. This value of S is much less than the theoretical value. Tests with a mineral oil known as "Viscolite" and other oils failed to show any superiority of fatty, as compared with mineral oils, or sucrose or glycerol solutions. Further tests were made with viscolite diluted with kerosene. A marked increase in friction, due to metallic contact, occurs at a higher value of S. In tests in which seizure occurred the maximum value of S when seizure occurred was about half its value at the point of minimum friction. The results of test show a higher friction with bronze bearing than with white metal. The material and smoothness of the bearing and journal have a greater influence upon the point of seizure than the nature of the lubricant. The friction of the white metal was 60 per cent. of that of an alloy of 89 per cent. of tin, 7½ per cent. of antimony, and 3½ per cent. of copper. The white metal bearing was reversed end for end after each increase of pressure, but it was impossible to raise the pressure above 429 lbs. per sq. in. When the end play of the journal was stopped the coefficient of friction was found to be increased by 50 per cent. after three days' running. The action of graphite is due to the replacing of the rubbing surfaces with new smoother surfaces, and adsorbed films from oils of good oiliness may have a similar effect. Numerous graphs show the results obtained in the tests. There is no evidence to show that the value of S at the point of minimum friction decreases as the oiliness improves. A journal friction-testing machine is not convenient for testing oiliness, as the clearance changes with wear. H. M.

Decolorising Lubricating Oils.—T. F. Ott. U.S.P. 1,448,084 of March 13, 1923. (Application May 1, 1920.)

Lubricating oil is decolorised by treating with acid, the acid being added in stages. After more than one addition of acid, water is added and the acid sludge removed. After the final treatment with acid and removal of sludge, the oil is washed with alkali. T. A. S.

Oil Distillation: for Lubricating Oils.—J. E. Schulze, Assr. to Red River Refining Co. U.S.P. 1,448,709 of March 13, 1923. (Application March 18, 1922.)

Mineral oil, containing no constituents as light as gasoline, is distilled under a pressure not exceeding 25 millimetres of mercury. From naphthenic base oil a fraction is obtained having a viscosity not less than 90 seconds (Saybolt Universal at 212° F.) and free from tarry matter. A lubricating oil having a viscosity not less than 150 seconds (Saybolt Universal at 100° F.), and further characterised by the fact that when distilled under a pressure of less than 5 millimetres of mercury the first and last 10 per cent. do not differ in viscosity by more than 700 secs. (Saybolt Universal at 100° F.). T. A. S.

Lubricating Oil.—E. G. Acheson. U.S.P. 1,448,246 of March 3, 1923. (Application Aug. 28, 1919.)

The lubricating oil has the composition and characteristics of an unfiltered distillate of lubricating grade, and a standardised colour and appearance due to the addition of a minute proportion of a deflocculated

body. Distillates of lubricating grade containing constituents removable by filtration are standardised by the addition of a minute proportion of a deflocculated body. T. A. S.

CENTRIFUGAL SEPARATION—METHOD AND MECHANISM FOR.—The Sharples Speciality Co., Assrs. to L. D. Jones. E.P. 191,750 of May 10, 1923. (Convention (U.S.) Oct. 7, 1921.)

When oil and a carrier liquid are delivered into a centrifugal rotor through a single feed pipe, considerable emulsification takes place. Portions of the carrier liquid remain emulsified with the oil, incomplete separation of wax also occurs and additional operations are necessary to secure a bright stock. In the present invention the carrier liquid is added separately at the discharge orifice communicating with the carrier liquid layer, and furthest from the axis of rotation of the rotor. By supplying the carrier liquid in this manner the apparatus is first filled with such liquid to the discharge level and additional quantities of such liquid are immediately discharged without causing any flow of the same through the rotor. Only such additions of carrier liquid are necessary as will replace the slight losses due to leakage and entrainment. Calcium chloride solution is the preferred carrier liquid when separating wax from chilled diluted cylinder stock. T. A. S.

COAL, SHALE AND ASPHALT.

CHEMICAL EXAMINATION OF THE ORGANIC MATTER IN OIL-SHALES.—R. H. McKee and R. T. Goodwin. *Quarterly of the Colorado School of Mines*, 1922, **18**, No. 1, *Supplement A*, p. 41.

In order to determine the chemical nature of kerogen four methods of procedure were employed—(*a*) chemical analysis of the organic content of shale; (*b*) the effect of heat on kerogen; (*c*) the action of solvents and reagents on shale; and (*d*) microscopic examination of the organic content of shale. The results of the ultimate analysis of a large number of different shales did not give any clue to the composition of the organic matter, neither did there appear to be any definite relation between the volatile matter as determined by analysis and the yield of oil by destructive distillation. For the examination of the effect of heat on kerogen the endothermic and exothermic reactions taking place during retorting were studied. In general it was found that endothermic reactions take place up to a temperature of about 500° C., and above that the reactions are exothermic, the two however nearly balancing in quantity. By retorting under a vacuum of about 8 m.m. a solid waxy material was obtained which was regarded as the intermediate product between kerogen and oil. By solubility in sulphuric acid it was entirely composed of unsaturated hydrocarbons, and by one distillation at ordinary pressure an oil was obtained with only 40–50% of unsaturated compounds. Hence it appears that a cracking temperature during retorting is necessary in order to produce saturated hydrocarbons. True solvents which have no chemical action on the kerogen were found to extract a merely negligible quantity from shales, but where such substances as sulphuryl chloride or selenium oxychloride were employed it was often possible after this treatment to dissolve 50% of the total volatile matter by means of alcohol. This effect, however, appears to be chlorination since the same results were obtained when chlorine itself was employed, showing also that the kerogen must be largely composed of unsaturated compounds. A

microscopic examination of different shales failed to reveal the presence of any oil as such. All the identifiable organic matter consisted of plant remains and no animal matter could be recognised. F. G. P. R.

BLOWN PETROLEUM PITCH.—D. Holde and S. Weill. *Petroleum*, 1923, **19**, 541–542.

Graefe having found that petroleum pitch blown at high temperatures gave higher saponification values than those generally accepted (8–21), experiments were made with three such blown pitches, of m. pts. 70°, 100° and 150° C. respectively, and a thin oil with a pungent odour, obtained in blowing the pitches. Such pitch is used as a covering material for roofing felt, and as a rubber substitute. Graefe found saponification values exceeding 60, and the oil from the blowing when treated with concentrated alkali partially coagulated, the coagulated portion giving a stringy mass when treated with mineral acid. As stearine pitch in benzene-alcohol solution gives a saponification value of 33–106, and lignite tar pitch a value of 20·7–32·4, the samples were tested as to their neutrality and saponification value in benzene-alcohol solution, using alkali blue 6 b as indicator. The pitches were neutral, and the oil from blowing nearly neutral. The saponification value of the pitch with m. pt. 70° C. was 12·05, with m. pt. 100° C. 39·9, with m. pt. 150° C. 42·0, and of the oil 17·3. The distillates obtained by distilling the pitches were dissolved in benzene-alcohol, and 1·2 to 1·8 g. was found to require 1 or 2 drops of N/10 potash solution for neutralisation, whereas similar distillates from stearine pitch show acid values of 5·2 to 20·3. The apparently greater saponification value of the pitches appears not to be due to the presence of esters, but to the oxidising action of the potash solution. H. M.

LOW TEMPERATURE COAL-TAR AND RESULTS OF SUPERHEATING.—F. Fischer. *Ber.* 1923, **56**, 601–3.

Tar obtained by the low temperature distillation of coal by Schutz (*J.C.S.* 1923, **123**, 1, 195) contained considerable amounts of benzol and phenol, unlike that obtained by the author. This is attributed chiefly to the form of retort employed by Schutz, wherein the volatile products are exposed to the highest temperature of the furnace (500–600° C.). It has already been shown that hydro-aromatic hydrocarbons are dehydrogenated to benzol, and phenols with a long side chain are broken down to phenol and olefinic hydrocarbons under these conditions. The volume of gas per ton of coal was also much greater than that usually produced by low-temperature carbonisation, which is additional proof of the occurrence of secondary decomposition.
F. G. P. R.

THE MARINE KEROGEN SHALES FROM THE OILFIELDS OF JAPAN.—J. Takahashi. *Science Reports of the Tohoku, University of Japan*, 1922, **1**. No. 2, 63–156.

The oil-yielding rocks of Japan belong to the Upper Tertiary period, probably Miocene and Pliocene; among which are found three species of shale—sandy in the uppermost horizon of the oil measures, with grey shale below and black shale in the lower horizon. These shales contain remains of marine organisms and some organic matter and yield oil on distillation. Detailed descriptions are given of different shales and methods of retorting, chemical character, nature and occurrence of

kerogen, etc. A difference between lacustrine and marine shales is found in the fact that a hard, elastic material resembling resin is precipitated by chloroform from a part of the extract from the former only. Terrestrial kerogen rock also yields on distillation a larger proportion of unsaturated hydrocarbons, probably owing to the larger content of resins. After a detailed description of shales from all countries the author concludes that the original matter contained in shales of a sapropelic nature is found in two principal forms—carbonaceous and bituminous—the latter having originated from the primary constituents of the shales and not being introduced from foreign sources after sedimentation. The kerogen of the Japanese oil-measure shales is considered to be different chemically from that of ordinary oil-shale to a great extent, and the Japanese oil measures are thought to consist chiefly of pyroclastic sediments intercalated with marine sapropelic shales containing a little kerogen. It is suggested that the oil originated to some extent from kelps which had been buried by volcanic detritus, but largely from sapropelic ooze after chemical change. The marine kerogen shales are nothing else but dehydrated and partially debituminised sapropelic ooze. The brine and bitumen thus squeezed out from the ooze migrated into porous beds and accumulated in pools. F. G. P. R.

Effects of Temperature and Time of Distillation on the Crude Oils from Oil-Shale.—L. C. Karrick. *U.S. Bureau of Mines Bulletin*, 1923.

Experiments were carried out to determine as accurately as possible the changes in quantity and quality of the oil produced by variations in the rate of heating oil-shale during retorting. The results showed that changes of quality may be due either to the temperature at which decomposition took place, or to the extent to which the thermal reaction had progressed. It was also conclusively proved that oil is actually produced at lower temperatures when the rate of distillation is slow than when rapid distillation is attempted. In either case initial formation of oil began at about 365° C. shale temperature, 25 per cent. of the total yield was off at 425° in the rapid distillation, and at 400° in the slow; 50 per cent. at 440° compared with 405°; 75 per cent., 400° to 420°; 100 per cent., 525° to 460°. Better quality oil is produced by slow distillation, owing to more complete decomposition of the "kerogen" within the, possibly, catalytic influence of the porous shale, and absence of carry over of undecomposed bitumen due to the velocity of the gases. At the slower rate, also, the bituminous substances exist for a longer period in the state of liquid and therefore afford a greater opportunity for low-temperature liquid-phase decomposition to take place, instead of cracking occurring in the gaseous phase, which is well known to favour production of unsaturated hydrocarbon compounds. F. G. P. R.

Bituminous Matter; Process of Distillation of.—W. Meischke-Smith, U.S. Pat. 1,450,327 of April 3, 1923. (Application April 26, 1920.)

Combustion is maintained in the lower portion of a vertical retort. The retort is fed with bituminous matter mixed with balls of uncombustible material. Combustion is so controlled that the bituminous matter is reduced to coke by the hot gases from the combustion zone before the balls and bituminous matter reach that zone. After leaving the bottom of the retort the balls are recovered and fed back to the retort with more bituminous matter. T. A. S.

IMPROVED APPARATUS FOR DISTILLATION OF OIL-PRODUCING MATERIALS.—Trent Process Corporation. Assees. of W. E. Trent. Brit. Pat. 174,336, of March 22, 1923. (Convention. (U.S.) Jan. 20, 1921.)

The material, such as coal, oil or mixtures of coal, oil, etc., is passed downwards through a structure consisting of a number of outer stationary hearths and a number of inner rotating hearths. The passage of the material is facilitated by the inclined structure of the hearths and by rabbles. The hearths are hollow and through them the heating medium is passed. As the apparatus works at low temperatures, the hearths are built of iron. T. A. S.

ASPHALT: APPARATUS FOR TREATMENT.—H. R. Wardell. Assr. to Central Commercial Co. U.S. Pat. 1,448,370 of March 13, 1923. (Application March 4, 1920.)

Molten asphalt is blown with a gas into which powdered material is fed. The apparatus consists of a horizontal cylindrical vessel, in which the asphalt is maintained liquid, fitted with a number of pipes through which the gas is blown. Powdered material in regulated quantity is fed into the gas stream from a hopper. T. A. S.

HYDROCARBONS FROM CARBONACEOUS SUBSTANCES SUCH AS COAL OR FROM DISTILLATION PRODUCTS THEREOF; PROCESS AND APPARATUS FOR MANUFACTURE OF.—H. Plauson and J. A. Vielle. Brit. Pat. 182,852. of July 5, 1922. (Application March 5, 1921.)

Hydrogenation of carbonaceous material is brought about by heating the material with hydrogen or water or both. If water is used the presence of oxygen in the reaction products shows that hydrogenation has taken place. The pressure to be used varies with the temperature; at 700° C. a pressure of 15-25 atmospheres is necessary, at 600° C. a pressure of 50–60 atmospheres, at 500° C. a pressure of 75–100 atmospheres, and at 400° C. a pressure of 150–200 atmospheres. The solid matter to be treated, *e.g.* brown coal, is mixed with oil and the mixture pumped through a heated tube, hydrogen being also admitted. The velocity through the tube is such that the passage of the material takes some hours, *e.g.* 4 hours to 400–450° C. The reaction tube is made of nickel or nickel steel. After leaving the tube the treated mixture is allowed to expand into an expansion vessel from which vapours are taken to fractional condensers. The unused hydrogen is returned to the apparatus. T. A. S.

OIL SHALE RETORT.—G. E. Holmes. U.S. Pat. 1,451,575 of April 10, 1923. (Application Dec. 27, 1919.)

A closed horizontal retort extends through a heating furnace and is subjected to graduated external heat. Means are provided for admitting shale to the cooler end of the retort and for agitating and conveying the shale along the retort. The distillation products are withdrawn progressively along the retort. Low pressure steam is injected. The plant works continuously. T. A. S.

SHALE; TREATMENT OF.—S. Marks. U.S. Pat. 1,451,367 of April 10, 1923. (Application Nov. 10, 1921.)

Ground shale is heated with agitation in the presence of dry gaseous products of combustion to temperatures in excess of 600° F. The shale and gases are then passed to a secondary heating zone where

the temperature is raised and superheated steam admitted until the evolved gases contain a predetermined amount of hydrogen. Combustible gas is obtained. T. A. S.

MATERIALS CONTAINING HYDROCARBONS; APPARATUS FOR TREATING.—C. P. Bowie and Mr. J. Gavin. U.S. Pat. 1,453,037 of April 24, 1923. (Application June 1, 1922.)

The material is fed into a conical retort, the bottom of which is heated. Off-takes for vapour are provided in the wall of the retort. The discharge of spent material is brought about by rotating rabbles which pass the material through a sealed discharge space at the outer edge of the bottom hearth. T. A. S.

IMPROVEMENTS IN PRODUCTION OF HYDROCARBONS.—The U.K. Oil Co. G. F. Forwood and J. G. Taplay. E.P. 197,069 of May 10, 1923. (Application Feb. 16, 1922.)

A retort containing shale is heated to 400° C. in the upper portion, and 600° C. in the lower portion. Steam is admitted in the upper portion and the volatile oil is carried down the retort, in the lower part of which cracking and hydrogenation take place. The volatile products are taken to condensers. Considerable amounts of permanent gas containing hydrogen are formed. It is found very advantageous to return these gases after freeing them from sulphuretted hydrogen and carbon dioxide to the upper portion of the shale retort. The presence of these gases prevents the oil vapours undergoing undesirable decomposition and assists in producing hydrogenation. Working in this manner less steam is required, and the products are more easily desulphurised. T. A. S.

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Abstracts.

Compiled by S. Bowman, A.I.C., P. Dvorkowitz, Ph.D., H. Moore, A.I.C., F.C.S., F. G. P. Remfry, B.A., D.Sc., A.I.C., and T. A. Smith, B.Sc., F.I.C.

General.

The Genesis of Petroleum.—E. Pyhälä. *Petroleum*, 1923, **19**, 535–539.

II.

The optical activity of petroleum has been regarded as a strong proof of its organic origin. Very few petroleum oils have been found which are not more or less optically active. Though it has been shown that animal remains, such as might be supposed to form petroleum, yield little active substances, cholesterol has been looked upon as an organic basis of petroleum. It has, however, been found impossible to detect cholesterol or its derivatives in petroleum, and the colour reactions of petroleum, supposed to be due to cholesterol, have been found to be common to other substances not containing cholesterol, as turpentine and lavender oils. Terpenes and allied hydrocarbons have been found in many varieties of petroleum, and menthane and hexahydrocumol have been isolated from Baku petroleum. The optical activity of petroleum tends to become concentrated in certain fractions, but may be removed by careful refining. The author has isolated an oil smelling of turpentine or lavender oil from the residuum of the alkali washing of lubricating oil. The residuum was neutralised with diluted sulphuric acid, and the oil obtained distilled *in vacuo*. A yellow distillate was obtained, which was washed with alkali to remove organic acids, and then shaken with sulphuric acid. From the acid solution, by diluting with water and adding alkali, a feebly optically active oil was obtained, smelling of turpentine or lavender oil, with a sp. gr. of 0.8570 at 18·25° C. A solution of the oil in chloroform, on treatment with sulphuric acid, gives a blood-red colouration, gradually fading into greenish-brown. The oil gives no cholesterol reaction, but similar reactions to those given by French turpentine or oil of juniper. Hydrocarbons of the terpene type were also obtained from the acid sludge from the refining of the kerosene fraction of Baku petroleum. These oils were feebly dextro-active. As optically active substances are found in petroleum only in minute quantities they may be accessory substances of vegetable origin, and may have been taken up by the petroleum after having migrated to strata where vegetable remains are abundant. H. M.

The Conversion of Methyl Chloride to Methanol.—R. H. McKee and S. P. Burke. *Ind. Eng. Chem.* 1923, **15**, No. 7, 682–8.

In pre-war days over 10 million gallons a year of methanol were made in the United States by the destructive distillation of hard wood. During the war competitive processes were developed, involving the production of acetic acid and acetone by the fermentation process. Methane from

natural gas, however, seems to provide a better raw material now that chlorination to methyl chloride, without the formation of higher chlorides, has been successfully accomplished (*Bur. Mines. Tech. Paper* **255** (1921), and Curme, U.S.P. 1,422,838 of 1922). The present paper deals with methods of replacing Cl by $-OH$. A résumé of previous work is first given, then accounts of the use of steam, aluminium, and calcium hydroxide as hydrolysing agents and finally a theoretical consideration of the mechanism of the reactions involved. Steam was employed up to temperatures of 350° C., and in ratios varying from 1 : 1 to 3 : 1, and with and without catalysts, but in no case was anything more than a trace of methanol formed. With aluminium hydroxide a small amount of methanol was formed at the beginning of the run, but the reaction soon ceased owing to decomposition of the hydroxide into oxide and water, which occurred even at temperatures as low as 200° C. With calcium hydroxide results were obtained which gave promise of commercial possibilities, the best yield of 67 per cent. of methanol being obtained at a temperature of 350° C. The methyl chloride was first bubbled through water, then passed through calcium hydroxide contained in a vertical tube furnace, and finally bubbled through water saturated with methyl chloride which acted as a very efficient absorbent. The original paper should be consulted for the theoretical considerations of the reaction.

F. G. P. R.

THE CAPILLARY THEORY OF ADSORPTION.—W. A. Patrick. *Chem. Met. Eng.* 1923, **29**, No. 1, 9–10.

The author stated that experiments on adsorption have always included capillarity as one effect, no experiments on plain surface adsorption having ever been done. The fundamental relationship of capillarity expressed in terms of vapour pressure, radius of curvature, surface tension, molecular weight, density, etc., is discussed, and in order to account for the lowering of vapour pressure produced in adsorption by silica gel it is necessary to assume pores of molecular dimensions. Experiments with SO_2, CO_2, and butane with silica gel gave a wide diversity of results, and in order to correlate these the extent of adsorption, *i.e.*, filling of the capillary pores of the adsorbent, must be expressed in terms of volume and not of mass. Using the basic formula of capillarity the above results can then be plotted on a single curve, which must be regarded as empirical. From the practical standpoint it is obvious that other forces besides capillarity would make silica gel retain gases, and theoretically the formula cannot be deduced from the basic thermodynamic relationships of capillarity. It can, however, be said that the physical properties of the liquid resulting from the condensation of the adsorbed gas are the determining factors in the extent of the adsorption. It is concluded that the surface tension of a liquid exhibiting capillarity curvature is not constant, and if a thermodynamic equation is calculated from the results of adsorption with the system SO_2—silica gel at various temperatures and pressures it is found that the two constants of the equation are not constant and that the radius of the capillary spaces are of molecular dimensions, and in some cases less than 0·2 m.m. in diameter. Thus the derived formula cannot be regarded as a workable one. Applying these ideas to dissolved substances it follows that the components of a solution that most easily wet the porous adsorbents will be adsorbed from solution, and it is thus possible to predict what component will be thus adsorbed by silica gel. In practice this can be done at least qualitatively.

F. G. P. R.

HYDROGENATED OIL FOR OIL BATHS.—G. R. Robertson. *Ind. Eng. Chem.* 1923, **15**, No. 7, 701.

"Hard" hydrogenated vegetable oil, the intermediate product of shortening, has proved to be far superior to heavy mineral oils for use in oil baths. Hard Sesame oil, approximated 90 per cent. hydrogenated, has M.P. about 60° C., open pan flash 320° C., but will not burn unaided below 350° C., and on resinification will not stick to either iron or glass, but cracks and falls apart. It is much cheaper than fusible metal. Hard hydrogenated cotton-seed oil has M.P. 60° C. and flash point 305° C., and is about as good as the Sesame oil. If water gets spilled into the bath it is easily removed by cooling the oil, cracking the solid and exposing to air. Fire risks in laboratories are largely eliminated by the use of such oils in place of mineral oils. F. G. P. R.

SAYBOLT VISCOSITY MEASUREMENTS AND THEIR CONTROL.—P. E. Klopsteg and W. H. Stannard. *Ind. Eng. Chem.* 1923, **15**, No. 7, 702–7.

In the A.S.T.M. specification for measuring viscosities by the Saybolt instrument it is stated that "the bath shall be held constant within 0·25° F. (0.14° C.)," and "that it is extremely important that the temperature of the oil in the bath be maintained constant during the entire time consumed in making the test." The authors show that this is impossible to do when stirring the oil in the tube by hand, as specified by the A.S.T.M., and attempting temperature control by the usual mercury in glass or metal type of thermo-regulator. They have therefore devised a special electrical heating coil and regulator, the latter being in the form of a bimetallic helix immersed in the oil bath, which operates a lever making contact through the heater coil. The bath has rounded edges and thorough circulation of the oil is obtained by means of a propellor. Charts are given of temperature variations in the oil bath during a run of nine days without attention, and during the whole time the maximum departure from the average temperature was less than 0·2° C. Stirring of the oil in the viscosity tube was best done by means of a helical screw attached to the stem of the thermometer which was rotated at 200 revolutions per minute. With a cover on, and the bath at 100·3° C., a perfectly steady temperature of 99·9° C. was obtained during 24 minutes, during which time no variation at all could be detected on the thermometer. Photographs of the apparatus and details of the design of the thermo-regulator are given. The authors conclude that without this, or a similarly efficient temperature regulator, it is quite impossible to comply with the requirements of the A.S.T.M. specification. F. G. P. R.

ON THE APPLICATION OF RADIOMETRIC SURVEYING METHODS TO PROSPECTING OF OIL DEPOSITS.—L. N. Bogozavlenaky. *Nepht. Slantz. Khoz.*, IV. (2), 239.

The investigation of radioactivity at certain points on the earth's surface is based on the varying concentration of radioelements in the earth's crust. After determining air conductivity at different points, curves connecting points of equal intensity (isorads) can be constructed, thus delineating the contours of a radioactive body. Accumulations of radioelements may be due to radioactive ores and minerals, to gases containing emanations, or to emanations dissolved in water or natural organic liquids, such as petroleum. Compared with water, oils possess a higher absorption power for radioactive emanations (at 25° C. the absorption power of petroleum is forty-five times that of water), so that

even in sedimentary rocks oils have a greater radioactivity than the enclosing rocks. Thus prospecting is simplified by determining the contours of oil deposits by the radioactivity of the earth's surface. P. D.

GROZNI PETROLEUM INDUSTRY.—*Grozni Petr. Ind. Journ.*, March, 1923.

The production of crude oil in Grozni from October 1st, 1922, to April 1st, 1923, amounted to 44,162,300 poods, of which 19,985,647 poods was paraffin-base oil yielding 10,185,795 poods of paraffin scale. Of the total production 43·5%, or 19,241,692 poods, was obtained from spouters. The number of producing wells was 229, and 40 drilling rigs were in operation, 12,000 feet being drilled during the six months.

P. D.

RESERVE OF PETROLEUM LANDS IN THE URAL DISTRICT.—*Grozni. Petr. Ind. Journ.*, March, 1923, p. 10.

At the present time the development of the petroleum fields of Russia is of great importance and, although the production in Baku and Grozni can be increased, attention will have to be given to new districts. Of these the Ural District, with an area of 70,000 sq. versts, is one of the most important. Geological investigations made by Prof. N. Tychonovitch have defined five petroliferous districts, each 150 versts long and 50 versts wide, as follows :—(1) Tamyrsh ; (2) Tarsakansh ; (3) Ymankaryush ; (4) Dossor ; and (5) Gouriev.

In the north-eastern part a number of asphalt deposits have been found, and in the south-western district liquid petroleum oozes out in several places.

At Makat and Dossor, where the oil-bearing strata are of considerable thickness, a heavy oil, sp. gr. 0·910, is obtained, while gas is met with in deeper strata. At Dossor, the first stratum is at a depth of 400 to 500 feet, and the second at about 700 feet, one of the chief features of this district being that the petroleum is between two water-bearing beds.

In spite of water difficulties, however, 16,800,000 poods of oil were produced in Dossor in 1916. Another district which has already produced oil is Guriev, where a spouter yielded 100,000 poods of light oil, sp. gr. 0·782 in two days from a depth of 805 feet. P. D.

WAGES OF WORKERS IN THE GROZNI PETROLEUM INDUSTRY.—*Grozni. Petr. Ind. Journ.*, March, 1923, p. 30.

The official table of wages paid in October, 1922, compared with 1913, shows the present position of all workers in the Grozni oilfields, which were nationalised, together with other associated enterprises, by the Soviet Government three years ago :—

	1922.		1913.
	Roubles.	Kopecks.	Roubles.
Manager	51	0	425
Accountant	33	0	275
Assistant Accountant ..	23	9	110
Mechanic	14	70	60
Fitter	16	80	55
Clerk	11	39	45
Baler	11	39	35
Workman	7	87	25

Taking into consideration the cost of the food starvation ration of 2700 calories at 4 roubles 6 kopecks per ration, it is apparent that the men cannot obtain sufficient food apart from other necessities of life.

P. D.

ON THE PHOTO-CHEMICAL PROPERTIES OF KALUJSKAJA PETROLEUM (MAIKOP). (Contribution to the methods of determining the "solid" asphalt.)—B. G. Tycinin. *Nepht. Slantz. Khoz.*, IV. (1), 73. 1923.

While extracting solid asphalt from Kalujskaja petroleum (Kuban-Black Sea district), after Holde's method, it was found that the solution in petroleum ether, free from asphalt, continued to deposit lake-like films on standing. This phenomenon is connected with the action of light, as in darkness the deposition of asphalt ceases, but begins again on exposure to light.

In the presence of large-surface solids (fine nickel wire, glass wool) it was found that more deposit was formed, the precipitation occurring principally on the surface of the solid introduced into the solution.

The following modifications of Holde's method are proposed:—

(1) The precipitation of asphalt to be made always in the dark.

(2) The introduction of solids, such as glass wool, to secure complete and quick precipitation.

The phenomenon can be explained as follows. The pre-formed asphalt is contained in the petroleum as a colloidal solution, and can be coagulated by the action of light, heat, or solvents. There is no chemical reaction, but only transformation of a pre-existing substance into another physical state. Thus the investigation of these substances is of importance for the elucidation of the formation and origin of petroleum. The following hypothesis of the formation of asphalt deposits is advanced. As petroleum rises to the earth's surface, the asphalt contained in it is coagulated by the action of light and deposited on soil particles, forming the nucleus of an asphalt deposit. The more volatile portions of the petroleum evaporate, the heavy parts are partly washed out, and partly remain with the asphalt nucleus, and further transformations (oxidation, polymerisation) result in the formation of "natural" asphalts.

P. D.

COPPER DISH AND "DOCTOR" TESTS NOT SO GOOD.—J. V. Meigs and E. J. Ford. *The Refiner*, 1923, **2**, Nos. 5, 6–7.

The writers consider that the present methods of testing gasoline, which generally consist of a distillation, copper dish and "doctor" tests, do not properly indicate the value of a motor fuel or the presence of compounds liable to be injurious to the engine. They have therefore made trials with various other reagents in an attempt to find one which will more nearly fulfil these requirements, and although no completely satisfactory results have been obtained, useful data has been accumulated which will assist in further prosecution of the work. The only criticism of the fractional distillation test lies in the fact that it is often considered so simple that little care is taken in carrying out the operation, and erroneous results are obtained. In the copper dish test confusion often arises between a gum deposit and actual corrosion of the metal surface, and it is recommended that the deposit should be well washed with ether or benzol to dissolve off the asphaltic matter before passing judgment on the amount of corrosion. Even with this precaution, however, it is often difficult to dissolve asphaltic and non-corrosive residues which have become baked on to the metal surface, and which may thus get unfairly classed as corrosive constituents of the sample. In order to overcome this the authors recommend employing a glass dish containing a granulated metal or metal turnings, since they have found it far easier to dissolve residues from metal in these forms than from a polished metal dish. Besides copper they have tried bismuth, cadmium, tin, antimony

and mercury. Of these bismuth was found superior to copper as an indicative test, and had the advantage that all residues were easily removed by ether or benzol, and in the case of a positive corrosive test the surface of the bismuth was left with a characteristic gunmetal colour after removal of the deposit. With regard to the "doctor" test, the authors have found it to often fail to indicate the presence of corrosive substances, and sometimes give a positive test with samples that were fit for consumption. The use of freshly precipitated white sulphur, in place of the usual yellow, appears to be advantageous and give better results. An alkaline solution of sodium stannite, as well as bismuth subnitrate, gave comparative results when shaken with samples of gasoline that were very promising. Ammoniacal silver nitrate was also tried for detection of aldehydes, and was found to give mirrors that compared in intensity with the indications given by the copper and bismuth. The authors conclude by saying that only a better knowledge of the chemical constituents of gasoline, especially unsaturated and sulphur compounds, will enable really useful tests to be devised for detecting the presence of impurities. F. G. P. R.

THE BITUMINOUS MARL OF ISMID IN ASIA MINOR.—F. Hansgirg and D. Sander. *Petroleum*, 1923, **19**, 576–585.

The bituminous marl occupies an area of 8·32 sq. km. on the northern slope of the high ground dividing the Gulf of Ismid in the north and the Gulf of Gemlik in the south. The hills formed a tertiary continental coast when the marl was deposited. No extension of the field is likely to the south and the west, but to north and east an extension is probable. A geological map of the marl district is given. The strata consist of a series of clay-marls 260 m. thick. The minute life that has caused the deposit of bitumen (sapropel and ostrakod) may be entirely absent from some members of the series, owing to the absence of proper settling basins at the time of their depositing, as at Jalakdere. The strata vary from horizontal to vertical. The richest bituminous deposit is an oil shale, with 22 per cent. of asphaltic content. Above and below the marl series is found a light yellow sandstone, containing many carbonised remains of plants. At Nuschedié and Hamidié a deeper and richer oil marl horizon is found. A typical section of the oil marl in Armutdere shows alternating beds of 1·2 m. oil marl (9·8 per cent. of bitumen), 3·5 m. without oil, 1·8 m. oil marl (11·3 per cent.), 3·2 m. without oil, 1·0 m. oil marl (10·5 per cent.), 3·0 m. without oil, 0·8 m. oil marl (12·8 per cent.), 5·0 m. without oil, 0·8 m. oil marl (9·7 per cent.), 5·0 m. without oil, and 1·0 m. oil marl of 16·8 per cent. The conglomerate overlying the marl is a difficult rock to drill, but it might probably be drilled by steel tools. The estimated amount of oil marl workable is 360 million tons. This amount may prove to be considerably more, but is not likely to prove less. The bituminous content varies from 5–20 per cent. The estimation of bitumen in the rock was roughly carried out on the spot by an apparatus, consisting of connected bulb and U tube; 5 g. of the powdered sample being placed in the bulb and heated, oil and water collected in the U tube, which was cut off and allowed to stand in a dessicator, the loss of weight showing amount of water, and the loss of weight after washing the tube out with benzene giving the oil content. The strata with 18–20 per cent. bitumen are generally only 0·5 m. to 0·7 m. in thickness. A sample of 200 kg. was collected over the whole field, and about 150 kg. distilled, and gave an average yield of 17 per cent. The gases evolved consisted of 48 per cent. carbon dioxide, 5 per cent. unsaturated hydrocarbons, 40 per cent.

hydrogen, 2 per cent. methane and 5 per cent. air, and had a calorific value of 5000 calories per cb. m. The oil is solid at ordinary temperatures, and contains much paraffin wax. The oil on standing deposited a good natural asphalt, the remaining oil having a sp. gr. of 0·84. On distilling fractions varying in sp. gr. from 0·82 to 0·91 were obtained, and 13 per cent. of residue. All the fractions had a high paraffin wax content. After freeing from paraffin some fractions of the distillate were capable of being made into high-class lubricating oil. The total products from the raw oil were, asphalt deposited before distillation, 2 per cent; petrol, 10 per cent.; lamp oil, 14 per cent.; gas oil, 18 per cent.; light lubricating oil, 12 per cent.; heavy lubricating oil, 15 per cent.; paraffin wax, 12 per cent.; asphaltic residue, 7 per cent.; and loss, 10 per cent.

H. M.

Results of Experiments on the Stability of Emulsions on Mineral Oils containing Varying Proportions of Rape Oil with Seawater.—Rosner and V. Navrat. *Petroleum*, 1923, **19**, 611–612.

Experiments were made to determine the minimum amount of added blown rape oil to mineral oils to produce a compounded oil, of which 25 c.c. mixed with 25 c.c. of seawater, and shaken for 10 min. in a cylinder of 100 c.c. capacity, should give an intimate emulsion, which should show little settling out of water after 24 hours standing. Trials were then made in seawater-cooled bearings, to test the emulsibility and the economy of consumption. The oil had to be free from sediment or other impurity, to correspond to specification as regards sp. gr., flash point, burning point, acidity, and viscosity, and it should not dry or become resinous. Mixtures were made of light distillate with 2–10 per cent. of rape oil of sp. gr. 0·927, Engler viscosity at 50°, 7·0 and flash point 243° C., and of heavy distillate with rape oil of sp. gr. 0·933, flash point 235° C., and viscosity 10·0. These mixtures gave emulsions which gave very little separation of water after several days standing. Further experiments were made with the addition of 0·3–1·6 per cent. of rape oil, and it was found that the addition of 1 per cent. rape oil was sufficient to ensure the formation of a permanent emulsion. Emulsions of equal quantities of oil and seawater appeared to be over-saturated, and oil separated out. Two parts of seawater and one part oil gave a permanent emulsion, but a greater proportion of seawater was unfavourable to emulsification.

H. M.

Domestic Burners for Oils.—J. B. Rathburn. *Petroleum Age* **10**, 11–12.

A description of the commoner types of burner is given, and the various errors in their design indicated. The principles on which all efficient and economical burners should be based are given in non-technical language, together with a consideration of the problem of air supply and combustion chamber design.

S. B.

Hydrocarbon Oils from Fats, Oils or Fatty Acids—Method of Manufacture of.—Nihon Glycerine Kogyo Kabushiki Kiaaha. E.P. 175,974 of Feb. 16, 1922. (Convention (Japan) Feb. 25, 1921.)

The sodium or calcium soaps of fatty acids are dried and distilled in an iron retort. In the presence of excess alkali distillation is carried on at 400–450° C., when the salt is neutral a temperature of 500–550° C. is required. From fish-oil a yield of 73–75 per cent. of hydrocarbon on the fatty acid used is obtained. Of the product 25 per cent. boils below 150° C., 55 per cent. between 150–300° C., and 20 per cent. over 300° C.

T. A. S.

Distillation and Refining.

European Practice in Cooling Chemical Liquids.—A. Thau. *Chem. Met. Eng.* 1923, **29**, No. 3, 98–102.

The author describes four types of coolers employed for the purpose of reducing the temperature of hot liquids, of which the first three are well known and in general use, whilst the fourth has only lately been brought to anything like practical shape. The first type described is that composed of horizontal tubes erected vertically above one another and connected at alternate ends by U bends. Cooling is obtained by means of a water-spray from a trough situated above the top tube, from which water trickles down from one tube to the next. The hot liquid enters at the bottom tube and leaves by the top, thus going counter-current to the cooling water. Sea water or dirty water can be used in this type provided a good flow of water over the pipes is maintained so that no deposit can remain, but with clean water the supply can be cut down to just sufficient to cover the whole pipe surface and evaporate completely. For this purpose a separate spray for each pipe is necessary. The second type of cooler is constructed like a water-tube boiler, the cooling water circulating through the nest of pipes whilst the hot liquor occupies the space between the tubes and the external shell. These are erected vertically above one another as in the first type and the hot liquid and cooling water flow in counter-current. These coolers are very efficient and can be easily controlled, but have the disadvantages that any deposit of solid quickly obstructs the passage of the liquid and leaks cannot easily be detected. The third type is very similar to the last except that instead of having headers for the liquid to pass from one tube to the next the whole of the cooling tubes are situated in a box, and by means of baffle-plates the liquid is made to circulate along the cooling tubes in an upward direction, the cooling water flowing counter-current. This cooler shares advantages and disadvantages with the last type. The fourth type is a direct-acting one in which the cooling water and liquid to be cooled come in direct contact and is, therefore, only suitable for oils and such like substances immiscible in water. The cooler is in the form of a box, having horizontal baffle-plates attached alternately to either end, by means of which the liquids have to follow a zig-zag path upwards or downwards. Depending on the specific gravity of the oil this is introduced at the top or bottom, and water at the opposite point. A gravity flow is employed and cooling takes place by direct contact of the two liquids. In using this cooler the oil must be fairly clean, otherwise emulsions are formed and separation made impossible. This type was first tried twelve years ago and, at first, did not prove a success, but cost of material after the war brought this cheaply-produced plant again to the fore and enabled the proper working methods to be elucidated. It has since then been erected and used with marked success in many places.

F. G. P. R.

Heat Losses at Refineries.—*Petroleum Age*, **11**, 1, p. 76–78.

An article dealing with losses commonly occurring in refinery practice and their prevention by means of efficient insulation.

The principal sources of heat losses are the brickwork settings, foundations and exposed portions of stills, *e.g.*, on a still setting of 17½-inch wall with an internal temperature of 2,000° F., 4½ ins. of insulation saves 31·5 lbs. of coal or its equivalent in oil per 1,000 sq. ft. per hour,

representing 60 per cent. of the heat lost or 33 per cent. of the fuel used.

Apart from the saving of fuel thus effected, proper insulation leads to a more uniform temperature, absence of local overheating, better fractionation, and increased capacity and life of the still.

S. B.

THE REGENERATIVE SYSTEM OF PETROLEUM DISTILLATION.—*Grozni Petr. Ind. Journ.*, March, 1923, p. 33.

Numerous patents have been granted for the utilisation of the heat contained in the hot vapours or residues leaving petroleum stills, and the chief method of regeneration of the heat is that used in Baku. Before entering the still the crude oil passes through a pre-heater where it is heated by the hot residue leaving the still. The utilisation of the heat from the vapours, however, has been adopted in only two refineries, one at Grozni and one at Fergan. In the latter the quantity of petroleum used for heating and loss is only 2½ per cent. The reason why the complete regenerative system is not more generally adopted is that it necessitates a very complicated system.

P. D.

GASOLINE RECOVERY FROM NATURAL GAS.—E. A. Starke. U.S.P. 1,439,921 of Dec. 26, 1922. (Appl. Sept. 12, 1921.)

Absorbing oil and hydrocarbon gas containing gasoline are passed into the suction of a pump. The oil-gas mixture is passed through a series of separators where the gas is further caused to take a tortuous path through oil. The non-absorbed gas is discharged, the enriched oil being taken to a still where the gasoline is removed.

T. A. S.

SLUDGE ACIDS: METHOD OF TREATING.—I. Hechenbleikner and F. J. Bartholomew, Assrs. to Chemical Construction Co. U.S.P. 1,457,030 of May 29, 1923. (Appl. June 15, 1922.)

Sludge acids or separated sludge acids are first internally heated to produce a partial concentration, whilst the carbon impurities are maintained suspended. The acids are then diluted to produce a separation of the impurities.

T. A. S.

CONDENSIBLE VAPOURS: METHOD OF SEPARATING FROM GASES.—C. R. Reed, Assr. to Hope Construction and Refining Co. U.S.P. 1,456,570 of May 29, 1923. (Appl. March 14, 1921.)

In the recovery of gasoline from natural gas two vessels containing absorbing liquid are used. Along the bottom of one of the vessels is a perforated pipe through which the rich gas is passed, permanent gas escaping at the top. The other vessel is a still. The two vessels are connected. A thermo-siphon is set up causing the absorbing liquid to pass through the still back to the absorbing vessel through a cooler.

T. A. S.

BLEACHING EARTH.—C. Cramer. U.S.P. 1,455,955 of May 22, 1923. (Appl. Feb. 27, 1922.)

Earths having excellent bleaching properties are obtained by mixing aluminium hydrosilicates with dilute hydrochloric acid and emulsifying the mixture intensely in the cold.

T. A. S.

LIGHT-COLOURED FATTY ACIDS—PRODUCTION OF.—J. W. Bodman, Assr. to William Garrigue & Co. U.S.P. 1,439,685 of Dec. 26, 1922. (Appl. Sept. 21, 1920.)

Dark-coloured fatty acids are evaporated, the vapours being mixed with a gaseous carrier. The mixed vapours are submitted to centrifugal action whilst being expanded and are also scrubbed during this treatment. The gaseous mixture is maintained above the vapourising point of the acids during these operations. The fatty acids are subsequently condensed. T. A. S.

GASOLINE FROM NATURAL GAS—RECOVERY OF.—E. S. Merriam. U.S.P. 1,457,786 of June 5, 1923. (Appl. Oct. 16, 1920.)

The gas is passed through an absorbing liquid which is subsequently distilled. The still is operated continuously, the absorbing liquid first passing through a separator in which the lightest hydrocarbons are removed as vapour. This vapour is passed through the liquid in the still to assist in the removal of the heavier hydrocarbons.

T. A. S.

CRUDE OIL AND GASES—PROCESS OF TREATING.—A. J. Paris. U.S.P. 1,405,153–4 of Jan. 31, 1922. (Appl.: (*a*) July 9, 1914; (*b*) May 25, 1916.)

(*a*) Gases other than air are used as carriers for petroleum vapours. The gaseous mixture is compressed and cooled to separate the condensable hydrocarbons.

(*b*) Hydrocarbons are recovered from gaseous mixtures by compressing the mixture and injecting a small quantity of heavy hydrocarbon oil to absorb the light hydrocarbons which are subsequently separated.

T. A. S.

ACID SLUDGE—PROCESS OF TREATING.—O. Bezanson, Assr. to Merrimac Chemical Co. U.S.P. 1,459,084, June 19, 1923. (Appl. May 14, 1921.)

Acid sludge is sprayed, with the necessary air into a chamber which is maintained at a temperature sufficiently high to decompose the sludge, its sulphur content being liberated as sulphur dioxide.

T. A. S.

DEHYDRATING PETROLEUM OILS—APPARATUS FOR.—F. W. Harris, Assr. to Petroleum Rectifying Co. U.S.P. 1,458,291 of June 12, 1923. (Appl. June 28, 1921.)

Emulsion is fed into a tank through a dehydrator containing two electrodes, the upper one being insulated. Arrangements are made so that the current passing between the electrodes is subjected to a coercive magnetic field.

T. A. S.

RECOVERY OF VAPOURS—PROCESS FOR.—E. A. Barbet. U.S.P. 1,452,778.

Vapours arising from reservoirs containing petrol, benzol, alcohol, ether, etc., are recovered by being passed up a column containing a suitable absorbent, the feed of the latter being automatically adjustable to the quantity of vapour mixture passing into the absorbent liquid. The absorbed vapours are then distilled off from the absorbent.

F. G. P. R.

APPARATUS FOR SEPARATING LIQUIDS OF DIFFERENT DENSITIES.—O. E. Hans. U.S.P. 1,450,545.

The vessel containing the two liquids is provided with a draw-off tube through which liquid from the lower portion of the upper, or lighter, liquid is drawn. In order to prevent entrance into the draw-off tube of the heavier liquid, a tube which can be maintained adjacent to the surface of the lighter liquid leads into the draw-off tube and thus maintains the latter full of the lighter only. F. G. P. R.

PETROLEUM OIL—APPARATUS FOR TREATING.—H. T. Maitland. U.S.P. 1,450,326.

The apparatus consists of a digester and a mechanical masticator wherein oil and washing liquid are brought into intimate contact. The masticator comprises a vessel within which is situated a rotatable upright hollow shaft to which is secured a hollow stirrer communicating interiorly with the shaft (the oil is introduced into the upper part of the shaft), passing thence into the stirrer and from there into the vessel, into which washing liquid is introduced through the bottom thereof.

F. G. P. R.

PURIFYING OILS.—J. Smith. U.S.P. 1,450,617.

Mineral oils are purified by digesting the oil under pressure with absorbent cotton waste saturated with alkali, in a steam-heated autoclave. F. G. P. R.

ABSORPTION OF GASOLINE FROM NATURAL GAS AND DISTILLATION GASES.—A. Von Groeling. U.S.P. 1,456,053.

In order to lessen the amount of lighter gases which are absorbed with gasoline the gas is compressed and allowed to impart its heat to one or more stills before entering the absorber, the latter being further cooled by expansion of some of the gas. F. G. P. R.

CRACKING.

OPERATING PRACTICE IN REFINING CRACKED PETROLEUM DISTILLATES.—G. Egloff and J. C. Morrell. *Chem. Met. Eng.* 1923, **29**, No. 2, 53–4.

The authors state that approximately 2,800 millions gallons of cracked gasoline will be produced during 1923 in the United States. The usual method of refining employed consists of acid washing, followed, or preceded, by a wash with sodium plumbite and a final distillation with steam. The prevalent idea that cracked distillates are not amenable to the same treatment as straight run spirit is incorrect, slight modifications of the ordinary practice being all that is necessary to obtain water white, sweet-smelling products. Agitation is performed by paddle, circulating pump or compressed air, the latter being most general though producing the largest losses. The continuous system of washing is strongly advocated in place of the batch method. Sulphuric acid of 66° Bé (95 per cent., sp. gr. 1·84) should be used as this is most economical and gives the best results, the quantity necessary varying from 1 to 7 lbs. per barrel of distillate. The least amount of acid possible should be used since the refining power is not proportional to the quantity, and an excess of acid produces a "burnt" product with a fixed yellow colour. The acid should be applied in several small doses, and is followed by a water and soda wash. In order to remove sulphur compounds a solution of litharge in caustic soda is usually employed, varying

in amount from 4 to 10 lbs. of litharge per 100 barrels of oil (125 to 150 lbs. litharge dissolved in 400–500 gals. of 12–13° Bé caustic soda for 1,400 barrels of oil). Some refineries treat with soda and then add litharge while agitating, but this is bad practice and wasteful in litharge. Sulphur should not be added at any stage after acid washing, though in the case of highly-sulphurous oils that have to be treated with litharge before acid sulphur may be used to aid precipitation. The authors outline four schemes of refining based on the above practice, which consist in (*a*) acid washing, (*b*) water wash, (*c*) caustic wash, and (*d*) distillation with steam. Litharge treatment, followed by a water wash, improves the product and should be applied directly before distillation, except in the case of highly-sulphurous oils when it should come before the acid treatment. A blended mixture of cracked and straight-run distillates is easier to refine than cracked distillate alone.

F. G. P. R.

Cracking Oils—Process and Apparatus for.—A. J. Mahan and T. J. Lynch. E.P. 197,576 of June 26, 1923.

Kerosene, under 3–6 lbs. pressure, is passed through a pre-heater to a cracking tube. Half of the cracking tube is maintained at 1292–1472° F. or part is kept at 1275° F. and a quarter of its length at 1500° F. The cracking-tube consists of 1¼-inch pipe, 18 ins. to 2 feet in length. After leaving the cracking-tube the products are passed to a condenser. A liquid suitable for use as a motor fuel or varnish solvent is obtained.

T. A. S.

Light Hydrocarbon Distillates—Production of.—D. W. Hovey. E. P. 197,372.

Oil is sprayed into a still heated by enclosed superheated steam or other heating fluid. A perforated open pipe delivers steam or inert gas to the bottom of the still. The vapours are taken from the still through a reflux condenser. The operation is carried out at 50–75 lbs. pressure. With higher pressures more gasoline is produced; with lower pressures kerosene results. The process is carried on until 60–75 per cent. of the contents of still have come over.

T. A. S.

Formic Acid—Improvements relating to Manufacture and Utilization of.—H. Nielsen and B. Laing. E.P. 198,385 of June 4, 1922.

The yield of condensible hydrocarbons obtained in the destructive distillation of materials such as peat is increased by the addition of salts of formic acid. The formic acid is prepared from methane-containing gas by passing it over a metallic-oxide catalyser or from carbon-monoxide by passing it with water vapour over a metallic catalyser. Suitable gases are air-gas, coal-gas, or natural gas. Activated copper sub-oxide prepared by roasting in air the copper obtained by precipitating copper sulphate with zinc is used as a catalyst. The reaction is endothermic and requires a temperature of from 250°–800° C.

T. A. S.

Partial Combustion Methods for Treating Aliphatic Hydrocarbons. C. P. Byrnes, Assee. of J. H. James. E.P. 173,750 of Dec. 21, 1921. Conv. (U.S.A.) Jan. 6, 1921.

The catalytic oxidation of hydrocarbons is described in E.P. 138,113. In the present invention the temperature of the reaction is controlled by dividing the catalyst into layers supported on screens and by diluting the reacting gases with steam or inert gas. High-boiling hydrocarbons containing unsaturated bodies are converted

by regulated partial combustion into motor spirit, aldehydes, aldehyde-acids, etc. The catalysts used are uranium and molybdenum oxides. The temperature of the layers of catalyst is determined by means of thermocouples and controlled as required by the admission of diluent. The oxidised products after the removal of motor spirit and aldehyde-acids may be used as lubricants for certain purposes. The viscosity of lubricating oil fraction may be increased by polymerising it at 200° C. in the presence of hydrochloric acid, zinc chloride or phosphoric anhydride. T. A. S.

PROCESS FOR RECOVERING CATALYST FROM CATALYSIS RESIDUE.—E. V. Owen, Assr. to the Hoover Co. U.S.P. 1,460,110 of June 26, 1923. (Appl. March 3, 1922.)

Aluminium chloride which has been used in the catalysis of hydrocarbons is recovered by causing the aluminium chloride-hydrocarbon compound to impinge, whilst in the liquid state, against a hot surface. The surface is maintained at such a temperature that the aluminium chloride is volatilised whilst the hydrocarbon settles out. T. A. S.

APPARATUS FOR CRACKING OILS.—W. B. D. Penniman. U.S.P. 1,459,515 of June 19, 1923. (Appl. March 10, 1917.)

Oil is cracked by heating it under pressure. The vapours from the cracking vessel are passed through a chamber containing a steam-coil where they are brought to a predetermined temperature and then taken to a condenser. The supply of steam is thermostatically controlled. T. A. S.

HYDROCARBON OILS—PROCESS FOR CONVERSION OF—INTO LOW-BOILING HYDROCARBONS.—W. L. Emerson. U.S.P. 1,414,400 of May 2, 1922. (Appl. May 10, 1920.)

A mass of heavy hydrocarbon-oil is distilled under pressure. The gaseous products are fractionally condensed in dephlegmators, part of the condensed liquids being returned to the still, whilst desired fractions are removed. T. A. S.

MANUFACTURE OF LIGHT HYDROCARBONS.—W. F. Rittman, Assrs. to Synthetic Hydrocarbon Co. U.S.P. 1,419,121-2-5 of June 6, 1922. (Appl. (*a*) May 18, 1915; (*b*) Feb. 5, 1917; (*c*) Aug. 3, 1917.)

(*a*) Hydrocarbon oils are cracked by passing them downwardly through a vertical unobstructed externally-heated chamber. The operation is carried out at cracking temperature and under pressures higher than atmospheric.

(*b*) Hydrocarbon oils are cracked in the liquid phase under pressure. The liquid is then vapourised and the vapours are heated whilst moving through a vertical tube to a temperature higher than that used in the liquid phase cracking.

(*c*) The hydrocarbon oil is vapourised by heating it near the surface; the rest of the liquid being kept cool. The vapours produced are heated to cracking temperature. T. A. S.

HYDROCARBON OILS—PROCESS FOR REFINING.—H. M. Lasher, Assr. to Kansas City Gasoline Co. U.S.P. 1,457,068 of May 29, 1923. (Appl. June 23, 1920.)

Hydrocarbon oil is heated to cracking temperature in the presence of a finely divided sodium chloride and under low pressure. T. A. S.

HYDROCARBONS OF LOW-BOILING POINT—PROCESS AND APPARATUS FOR PRODUCTION OF.—J. C. Black. U.S.P. 1,456,419 of May 22, 1923. (Appl. June 20, 1922.)

Hydrocarbon oil is passed through a series of heating coils, the temperature being raised in the last coil to that at which cracking takes place. After leaving the coils the oil passes to a chamber where carbon is precipitated owing to a reduction in the rate of flow of the oil. Cracking takes place under pressure. After leaving the carbon-depositing chamber the pressure on the oil is released and it enters a fractionating column where it is separated into hydrocarbons of different boiling points. T. A. S.

MANUFACTURE OF GASOLINE.—R. F. Bacon and B. T. Brooks, Assrs. to Gulf Refining Co. U.S.P. 1,439,683 of Dec. 26, 1922. (Appl. Oct. 19, 1914.)

Petroleum oil having a specific gravity of over 0·860 and free from heavy constituents is distilled and cracked under 60 to 300 lbs. pressure and at temperatures ranging from 300 to 500° C. T. A. S.

AROMATIC RING COMPOUNDS—CONVERSION OF—INTO MOTOR SPIRIT.—A. S. Ramage, Assr. to Chemical Research Syndicate. U.S.P. 1,439,976 of Dec. 26, 1922. (Appl. Jan. 28, 1921.)

Aromatic hydrocarbon vapours mixed with non-aromatic hydrocarbon vapours and with steam are passed over an iron compound heated to 530° C. or over. The iron compound is maintained in the ferrous state. T. A. S.

HYDROGENATING HYDROCARBONS.—H. Rostin. U.S.P. 1,451,052.

Unsaturated hydrocarbons are hydrogenated by means of vapourising the hydrocarbon and reacting on the vapours at a temperature of approximately 300° C. with hydrogen sulphide in the presence of a metal capable of combining with sulphur, whereby nascent hydrogen is liberated and combines with the unsaturated hydrocarbons. F. G. P. R.

PRODUCTION OF ANHYDROUS ALUMINIUM CHLORIDE.—B. S. Kirkpatrick U.S.P. 1,455,005.

An alloy of aluminium is made with a metal having a melting point lower than that of aluminium but whose chloride is formed at higher temperatures. Chlorine gas is passed into the molten mixture, when aluminium chloride distils off but the other metal remains substantially unchanged. In an alloy of 25 per cent of cadmium and 37·5 per cent. each of zinc and aluminium the chloride of the latter is formed at 200° C. F. G. P. R.

MOTOR FUELS.

THE CRUDE OIL AERO ENGINE.—A. E. L. Chorlton. A paper read before the International Air Congress, London, 1923.

Crude oil, or more accurately, residual oil in addition to being much cheaper has many advantages over petrol as a fuel for aero engines. It high flash-point reduces the fire hazard and the higher compression ratios possible increase the fuel economy. The high pressure used in feeding the fuel renders the supply to the engine more positive than when it depends on small heads and the elimination of the ignition system would remove a source of unreliability. As only air is compressed an economical two-stroke engine with its advantage of more even torque

for a given number of cylinders becomes possible. Various types of fuel injection are discussed, *e.g.*, air-borne (Diesel), air-less (pump discharge) and gas-borne (explosion injection) and drawings of the types of nozzles given. Turbulence is an important factor in promoting combustion, but also promotes heat loss, and its control is, therefore, important. The modern aero-engine designer considers an engine weight of 2 lbs. per b.h.p. desirable, but the author thinks that with the advantages of heavy oil this might be raised to 4 lbs. per b.h.p. The Beardmore 600 b.h.p. engine runs of Mexican fuel of 0·9 sp. gr. and weighs approximately 3 lb. per b.h.p. T. A. S.

KEROSENE AS MOTOR FUEL.—*Oil Age*, **19**, 3, p. 16.

Experiments have been carried out at the Carnegie Inst. of Technology, Pittsburg, to determine the relative efficiency of kerosene and oxidised kerosenes as motor fuels. Oxidised kerosenes are manufactured by catalytic oxidation from low-grade petroleum and although possessing a lower calorific value they have practically the same power-development as kerosene owing to more complete combustion. They are also less liable to detonation than straight kerosenes and eminently suitable for kerosene engines and for blending with petrol. S. B.

ANTI-KNOCKING COMPOUND.—*Nat. Petr. News*, **14**, 51, p. 34–35.

A compound known as tetra ethyl lead is claimed by T. Midgley, Junr., to possess properties which eliminate the detonation tendency of gasoline.

The structure ascribed to this compound is $Pb(C_2H_5)_4$ and is said to be made from alcohol and lead. It is a water white oil sp. gr. 1·62 and infinitely soluble in gasoline. Left on the skin for some time it stings slightly but gives rise to no eruption or permanent burn. It is decomposed slowly on exposure to sunlight, a white precipitate settling out which turns brown and then to metallic lead.

Apparently 3 c.c. of this substance added to a gallon of petrol is capable of increasing the efficiency of an engine 100 per cent. by allowing the use of higher compression and heavier spirit. S. B.

SOUTH AFRICA'S MOTOR FUEL.—*Petroleum Times*, 1923, **9**, No. 234, 968.

A motor fuel, consisting chiefly of alcohol obtained from the prickly pear, has been successfully tried out against the best quality petrol, and is said to be able to be produced to sell at 2s. a gallon, as against 3s. 7d. of petrol. Thirteen gals. of alcohol can be obtained from a ton of this fruit, the average crop of which is said to be at least 10 tons per acre. Prickly pear cactus at present covers about 2,000,000 acres in the Union and is an ever-increasing menace requiring a large expenditure of money to combat. This area could provide 260 million gallons of alcohol on the above estimate, which is about 22 times the annual consumption of the whole of South Africa. Queensland is also greatly affected by the prickly pear having 27 million acres covered by this pest, and New South Wales 60 million acres. F. G. P. R.

ALCOHOL—PETROL FUEL MIXTURES—PROCESS FOR STABILISING.—Ricard, Allenet et Cie. E.P. 191,000 of June 14, 1923.

Alcohol-petrol mixtures are prevented from separating into two layers on cooling by the addition of normal butyl alcohol. The addition of 4·75 volumes of normal butyl alcohol to 100 volumes of petrol (sp. gr. 0·729) renders the petrol miscible in all proportions with a mixture of equal parts of alcohol and benzol and the mixture remains homogeneous at 0° C. T. A. S.

FUEL FOR INTERNAL COMBUSTION ENGINES.—J. D. Riedel, Akt. Ges. Anses. of P. Friesenhahn.

Fuels consisting of hydronaphthalenes mixed with aliphatic hydrocarbons are improved by the addition of ethyl alcohol (methylated spirit) which prevents the formation of soot. A considerable increase in energy is obtained. The sensitiveness of the mixture to water and temperature is overcome by the addition of a small quantity of ether or high-boiling alcohol. A mixture of tetrahydronaphthalene 30 pts., light petroleum 35 pts., methylated spirit 30 pts., and amyl alcohol 5 parts remains perfectly limpid at temperatures as low as −10° C. and the equilibrium is not disturbed by the addition of a small quantity of water. T. A. S.

LUBRICATING OILS AND WAXES.

VISCOSITY OF LUBRICATING OILS AT LOW TEMPERATURE.—M. Okochi and M. Majima. *Rikwagaku Kenkyujo Iha*, 1923, **2**, 23–37. *J. Chem. Soc.*, 1923, **124**. aII. 463.

Viscosities were determined at temperatures ranging from 27° C. to −18° C. of rape seed, gingell, refined rape seed, camellia, mobile, soja bean, spindle, whale, olive, and machine oils, as well as of petroleum, liquid paraffin and some mixtures. The oils were forced through a glass capillary tube 10 cms. long by means of compressed air or suction and viscosities calculated by the formula $\eta = \pi R\ 4/8\ Vl\ (Pe - Pi)$, where R = radius of the capillary, V = volume of oil passing through, l = length of capillary and Pe and Pi the pressures of the oil at the exit and entrance of the capillary. Tables are given of the results. The increase of viscosity with the decrease of temperature is almost expressed by a hyperbola, and the formula $\eta = (a/T - b)^3/a$ is proposed, a, a and b being constants. The values of η calculated from this formula are almost identical with the experimental results except in the cases of gingell, soja and whale oils, where they do not coincide at low temperatures owing to the formation of solid particles. With petroleum the increase of η with lowering of temperature is very small, and addition of petroleum to other oils produces a similar result.

F. G. P. R.

VISCOSITY OF LUBRICANTS AT HIGH PRESSURE AND TEMPERATURE.—M. D. Hersey. A paper read before the International Air Congress, London, 1923.

In an internal combustion engine the temperature and pressures of the oil film in the connecting rod and crank shaft bearings, and particularly in the cylinders, must be far higher than are usually employed in routine testing. The pressures must be at least as high as the explosion pressure, whilst the corresponding temperature may equal or exceed the fire point of the oil. Though these temperatures are unknown it is evident that in comparing oils it is desirable to have experimental data on the absolute viscosity of all lubricants at the highest pressures and temperatures available. Two fixed and three mineral oils were experimented on at temperatures ranging from 22° C. to 100° C., and pressures ranging up to 4000 kg/cm² (about 50,000 lb./in²). In plotting the curves at each temperature the ordinate was the ratio of the absolute viscosity, at the stated temperature and pressure, to the absolute viscosity at the same temperature but atmospheric pressure, and was called the relative viscosity. Pressures were given on the

abscissa. The apparatus consisted of a rolling ball viscometer (*Proc. Amer. Soc. Test. Mat.*, 1914, **14**, 565) modified for use at high pressures. The first experiments were on lard and mineral oils at 200 kg./cm². quantitatively, and 500 kg./cm² qualitatively. Later Hyde published results on four fixed and four mineral oils at 1200 kg./cm². at 40° C. in which the conclusion was reached that the effect of pressure on the viscosity of fixed oils, as a class, is less than on mineral oils. At the higher pressures and temperatures employed by the author this distinction tends to disappear. The results obtained show that the relative viscosity curves at low temperatures for mineral oils ascend very steeply all the way, whilst with fixed oils there is a preliminary gradual slope. At higher temperatures, however (100° C.) both classes show a much more gradual and sweeping increase. F. G. P. R.

Sulfo-Acid Bodies in Lubricating Oils.—G. L. Oliensis. *Ind. Eng. Chem.*, 1923, **15**, No. 7, 690–3.

On treating lubricating oils with sulphuric acid a very complicated set of reactions takes place, the more important of which may be divided into three categories. (1) Diolefines and similar unstable hydrocarbons are mostly carried down as an insoluble sludge, though part of this may remain in suspension or on the walls of the agitator and on subsequent neutralization produce oil soluble colloids very favourable to the formation of water-in-oil emulsions. (2) Monoolefines and aromatic hydrocarbons form complicated and unstable alkyl sulphuric acids and esters many of which are soluble in oil and are only partly carried down in the acid sludge. On treatment with alkali a large portion is certainly still retained in the oil as neutral esters and alkyl sulphates which may be soluble in both oil and water. (3) Aromatic and other hydrocarbons in the presence of fuming acid form (*a*) sulphonic acids giving water soluble alkali sulphonates and (*b*) naphthenic acids soluble in oil in the acid state and in water when neutralised. They are genuine water-soluble soaps which tend to form oil-in-water emulsions. Thus after treatment an oil may contain a formidable array of oil and water soluble colloids, soaps, etc., though in such small amounts as to be practically impossible to detect by chemical means. During neutralization when persistent emulsions sometimes get formed it has been found that the addition of a small quantity of a water-soluble soap and agitation with steam will often successfully break the emulsion, indicating that the bodies responsible were oil-soluble emulsifying agents, such as neutralized sludge bodies. This reaction has been taken advantage of in two current demulsibility tests—the "Vacuum steam test" and the "Conradson Demulsibility test."

Vacuum steam test.—About two inches of water are put in a pint or quart milk bottle, one inch or so of oil added and agitated with open steam. Stand aside for about half an hour and then note appearance of emulsified layer between the oil and water. If none, or the layer is of a foamy bubbly character, the oil is supposed to be of good quality. If thick, creamy and homogeneous in appearance it is called "slop" and adversely reported on. This "slop" is supposed to be due to traces of sludge left in the oil, through insufficient treating. The rapidity of separation of the layers is not especially noted, and this test is the only one which judges by the appearance of the emulsified layer.

Conradson test is far more precise but basically the same. 100 c.c. of oil and 20 c.c. of distilled water are placed in a 200 c.c. graduated

cylinder and agitated with open steam for 1·0 mins. The cylinder is then placed in a water bath maintained at 130° F. and the appearance and volume of the oil, emulsion and water layers noted from time to time during one hour. The actual demulsibility figure, however, is not got from the speed of separation or the appearance of the layers, but by subtracting the percentage of water retained in the oil layer at, the end of one hour, from 100. The method of reporting results is (*a*) the volume of the three layers after each of the first five mins. and at the end of 10, 30 and 60 mins., giving the speed of demulsibility. (*b*) The appearance of the three layers is also noted and indicates the quality and purity of the oil. The consistency of the emulsion layer does not indicate a difference in kind, as stated in the Vacuum test, but has been proved by the author to be entirely due to differences in quantity of emulsion forming bodies present in the oil. The amount of emulsified layer is not however proportional to this quantity because viscosity plays a part in the speed of breakdown. To prove whether an increased emulsified layer is due to high viscosity or the presence of large amounts emulsifying agents a repeat test is done on the oily layer that separates out above the emulsion. If the same results are obtained then viscosity is the responsible agent. The differentiation of oil-soluble from water-soluble emulsifying colloids can be made by observing the appearance of the dividing line between the emulsified layer and the oil and water layers. If the former only are present the upper dividing line will be sharp and the lower wavy and indefinite, while the reverse occurs in the case of the latter. Where both are present both upper and lower division are indefinite. A rough idea of the quantity can also be got from the texture and density as well as the quantity of the emulsion layer. The value of the test is in detection of the presence of impurities, even if these may be considered advantageous as in the case of oils blended with fatty oils and soaps. Two theories of the cause of breakdown in oils have been advanced—cracking and oxidation. The author believes that both take part in the formation of sludge and gummy asphaltic compounds, the cracked oil probably acting as a starting point or catalyst for oxidation. The notoriously unstable sulphuric esters and alkyl sulphates which tend to break down even in storage at atmospheric temperatures certainly play a part in the formation of tars when subjected to high temperatures, and during storage are probably the cause of the development of acidity. The Conradson test is therefore of use in detecting the presence of such bodies as would be liable to promote breakdown of a lubricating oil under practical conditions and hence is of service both to the refiner and user. F. G. P. R.

THE CHEMICAL AND PHYSICAL PROPERTIES OF INSULATING OILS.—Report from Brit. Electrical and Allied Industries Research Association. *J. Inst. Elect. Eng.*, 1923, **61**, 661–674.

In selecting methods of testing the quantities measured should be, as far as possible, chemical or physical constants, and where the quantities vary with the conditions of testing, the conditions should correspond as closely as possible to working conditions. There appears to be no immediate possibility of the utilization of coal-tar distillation products and shale oils as sources of insulating oils. Colour cannot be taken as an indication of the sludging characteristics of an oil. Trouble from oil in transformers is generally due to the presence of air, which oxidises and polymerises the oil, causing deposits to settle on the windings, and over-heating. Transformers should be designed to

exclude air. Oils used for the sludging test were from Burmah, Russia and from the U.S. Pale Russian oils were found to be the best, but some American oils were equally good. The sludging was always accompanied with darkening in colour. It was found best to use air instead of oxygen in the test, as with oxygen varnish-like deposits, difficult to wash and dissolve, were formed, whilst with air the deposit was granular, and the air test corresponds best with actual conditions. A special form of reaction flask was used, with electric heating of bath, thermostatic control and mechanical stirring. The flasks must be joined up to the air supply in parallel, and the air supply accurately measured. The Kissling test is not a good indication of the suitability of oil for use in transformers, nor can tintometer tests be relied upon. The Michie and the B.T.H. evaporation tests were found satisfactory, and gave an indication of the economy and safety of the oil in use. The flash point test showed considerable difference in the results of different experiments. Solvents used to clean the cup must be carefully driven off before making a new experiment. Tests of viscosity, taken at temperatures of 15·5°, 50° and 80° C., gave fairly concordant results between the various experimenters. Tests for acidity and alkalinity are precautionary to ensure the use of well-refined oils. They also indicate the formation of acid during sludging. The iodine absorption tests show distinct relationship with the practical sludging of the oil, especially when air is employed, and give a rough but rapid means of estimating the sludging propensity. In the density tests the small disparities were due to incorrect temperature control. The determination of the coefficient of volumetric expansion was attained by the making of accurate specific gravity tests at varying temperatures. The use of quartz vessels leads to greater accuracy. For the cold test (solidification) the Archbutt method is recommended. The specific heat was determined by the electrical method, the oil being stirred throughout the experiment. The specific heat value is not necessary for the drawing up of a specification. The specific heat of unit volume of each oil has a constant value of 0·390 at about 15·5° C. and of 0·405 at 50° C. The latent heat of vaporization was determined in a specially designed electrically heated vessel, in which oil was vaporized, and the results were checked by measuring the heat given out when the oil vapour was condensed in a calorimeter. To determine vapour pressures a little of the oil was introduced into the vacuum above a barometer column, the depression of the column at a certain temperature noted, and the result corrected to 0° C. In no case was the vapour pressure at 150° C. greater than 70 mm. mercury. The action of the catalysers on oil at 100° C. with 500 hours' heating was tested. It was concluded that dryers containing lead or cobalt, or manganese resinates or acetates, are liable to increase the quantity of sludge formed by an oil. H. M.

CHLORINATION OF MONTAN WAX.—Firma H. Stinnes, comm. to H. K. Jennings. E.P. 199, 073.

Crude Montan wax as obtained by extracting liquids with solvents is dark coloured and brittle and hence has as little technical value. Treatment with chlorine gives products more like resin. In the present process the operation is carried out in the presence of water. Finely divided wax, suspended in 10 per cent. caustic soda or milk of lime, is treated with nascent chlorine at 85–95° C. until the solution gives an acid reaction. The wax is converted into light brown wax-like substance which is stable. The same products are obtained if the

wax is treated with hydrochloric acid and potassium chlorate. The consistency of the product is dependent on the proportion of chlorine used, becoming softer as the quantity of chlorine is increased. T. A. S.

Shale, Coal and Asphalt.

The Low Temperature Carbonisation of Coal.—H. H. Curtis and W. A. Chapman. *Chem. Met. Eng.* **28**, 11–17.

The development of the Carbocoal Process for the manufacture of smokeless fuel by means of low temperature carbonisation is described. The original scheme of this process was to remove only such part of the volatile matter as would leave the residue in a plastic condition suitable for briquetting. Owing to the innumerable difficulties encountered in the working of retorts the scheme had eventually to be modified so that carbonisation was taken past the plastic stage to a residue suitable for grinding and subsequent briquetting with a binder.

The first type of retort employed consisted of a stationary horizontal cast iron cylinder 3 ft. in diameter and 17 ft. long, made in two sections, with a single paddle shaft through the centre. This shaft consisted of an 8-in. wrought iron pipe, with gudgeons for bearings in each end, and carried about forty paddles set at such an angle as would both stir the coal and advance it through the retort. Crushed coal was fed into the retort by means of a screw and the plastic residue discharged in a similar manner.

This type of retort soon proved itself to be a failure owing to the fusing coal baking itself on the stirring mechanism and eventually plugging the retort with a mass of semi-fused material, resulting in a breakdown in the machinery. Nine different types of retorts were tried in turn, each designed to meet some difficulty previously experienced, before it was realised that production of semi-plastic residue was unfeasible.

Evolution in design culminated in the Clinchfield retort which consisted of a muffle heart-shape in section, made of carborundum, 7 ft. diameter and 16 ft. long. Although possessing better heat transfer the metal soon warped and became useless.

The muffle is supported in fireclay saddles and heated by two rows of ten open pipe burners supplied from a gas tunnel underneath the retort. Stirring mechanism consisted of twin paddle shafts of cast steel.

The discharged carbon residue containing 10 to 12 per cent. volatile matter is a light friable semi-coke and is ground to powder by means of ball mills, mixed with a binder and pressed into briquettes. These have to be finally baked in an ordinary gas retort in order to get rid of the pitch used for binding and so render them smokeless. S. B.

Shale Oil Unsaturates.—C. W. Botkin and R. W. Boyd. *Petroleum Age* **10**. No. 11, 78–85.

Previous work by one of the authors (Botkin) has shown that crude shale oil contains a high percentage of constituents soluble in cold concentrated sulphuric acid, but on distillation yields lighter oils containing a much lower percentage of acid soluble constituents. In order to obtain a more complete knowledge of the properties of these oils iodometric methods have been applied in conjunction with sulphuric acid absorption figures, having regard to the fact that the latter is not a true measure of unsaturation.

As a preliminary it was necessary to investigate the effect of the different "iodine value" reagents and experiments were carried out using the Hubl and Hanns methods on crude and once-run oil, varying the excess of reagent in all cases. It was found that the iodine value of shale oils increased with the excess of reagent used in both methods, but the variation shown by the Hubl method was the least, and therefore was adopted for the purpose in view, especially in view of the fact that this reagent was practically unabsorbed by aromatics present. It was also shown that the iodine value of the crude shale oil, so determined, was only slightly higher than the once-run oil, although the former was much more soluble in cold 96 per cent. sulphuric acid. The light acid soluble oil formed by the decomposition of shale oil appears to be saturated to the iodine reagent.

The sulphuric acid absorption and iodine value, and nitrogen content, were determined for each 10 per cent. fraction of a crude and once-run distillate. Acid soluble components and nitrogen content increased with the gravity, whereas iodine values decreased.

The light acid-soluble oil contains over 1 per cent. of nitrogen, and it is suggested that this is responsible for gum formation, and the ratio of sulphuric acid absorption to iodine value may serve as an indication of the presence of these constituents. S. B.

Motor Spirits and the Lignite Industry.—E. Marcotte. Paper read at the International Air Congress, London, 1923.

In the present state of aviation petrol appears to be the best fuel for internal combustion engines, especially at high altitudes. The addition of benzol decreases the tendency to preignite and should economise aerial transport if obtainable in sufficient quantities and at a low enough price. Up to the present low temperature carbonisation of coal has been the only source of supply and is insufficient in France to replace the motor fuels now used. The carbonisation of lignite could, however, make up the difference. One ton of lignite containing 15 per cent. of moisture will give 600 kgs. coke for briquetting, 12–15 litres of benzol and 50–80 kgs. of tar from which burning oil, phenols and lubricating oils can be got. 100 carbonisation and distilling works, capable of treating 1000 tonnes of lignite a day, would produce 5000 tonnes of benzol, which would suffice for the needs of the whole of France. The Tozer retort which has been used on the lignites of Laluque has given good results, whilst the distillation process of Dr. Duplan produces a yield of 90 per cent. of valuable products from the tar. In this process 50 per cent. of coke, as a catalyst, from the preceding distillation is mixed with the tar which is distilled from flat bottomed stills having a large heating surface. Coal tar products become congealed at low temperatures on account of the presence of naphthaline, but this disadvantage is absent in lignite oils which contain paraffins only. The products are therefore suitable for use in internal combustion engines, and it should be of interest to study methodically the best conditions for their use. F. G. P. R.

The Utilisation of Nitrogen contained in Russian Shales.—By V. K. Valgis and M. Magurd. *Nepht. Slantz. Khoz.* **4**, (1), 88, 1923.

This investigation had as its object the collection of the scattered data on the quantity of nitrogen in Russian shales, and of undertaking experiments with two samples of shale from Weimarn and Volga to determine the distribution of nitrogen in the products of their dry distillation

and the conditions giving maximum percentage of ammonia. The Weimarn shales were poor in nitrogen—average content 0·2 per cent.—and it was unprofitably distributed in the products, the aqueous solution containing only 5·5 per cent. of the total. The nitrogen content of Volga shale averaged about 0·4 per cent., of which 18·9 per cent. passed into the aqueous solution on dry distillation, a higher percentage than given by Scottish shales (17 per cent.). As with Scottish shales and other combustibles, distillation with steam, or with steam and air, has a favourable influence on the quantity of ammonia given off by Russian shales. P. D.

Some Methods of Analysis of Oil-Shales and Sapropelites.—By M. F. Strunnikov. *Nepht. Slantz. Khoz.* **4**, (2), 276.

The necessity of changes in methods of shale analysis, and of applying new methods, is demonstrated by the work accomplished in the last five years in the organic technology laboratory of the Polytechnic Institute in Petrograd under V. K. Valgis.

Hygroscopic and general moisture is determined in an exsiccator, or in a drying oven at 90° C., with a powdered sample. The shale undergoes a perceptible oxidation in a drying oven at 100–105°, but at 90° this is much less. During drying the shale (1) loses water and carbonic acid, and (2) absorbs the oxygen of the air. Drying is stopped when the weight begins to show an increase or remains constant, the loss of weight calculated from the lowest weight obtained corresponding to the amount of moisture.

Volatile substances are determined by Muck's method. To obtain concordant results, about 0·5 g. of the shale must be taken and the platinum crucible employed must be 4–5 cm. high. As the mineral part of shale contains carbonates and ferrous iron, the ash determination comprises (1) combustion of organic matter and decomposition of carbonates (loss of weight), and (2) formation of ferric iron (gain of weight). Calcination in a platinum crucible is continued until the weight increases or is constant.

Nitrogen content is calculated as an average of determinations by the methods of Kjeldahl and Dumas.

Sulphur content is determined after Eschka, but 0·5 g. of shale must be taken if volatile matter is of large amount. A definite relation is observed between specific weight of shales from the same deposit and their volatile content, allowing control of output at the mine where facilities for determination of volatile matter are not always available.

The specific weight is obtained by the Chatelier-Kandlo apparatus used in the cement industry, water, turpentine, or benzine being used as liquid.

All the methods are applicable to sapropelites, but moisture is more quickly determined in an evacuated exsiccator than in a drying oven, both methods giving equal results. P. D.

Oil Shales: Process and Apparatus for Treating.—W. T. Kirby. U.S.P. 1,458,983 of June 19, 1923. Appl. Sept. 19, 1921.

Finely divided shale, mixed with a high boiling oil, is pumped through a tube heated to a high temperature to a vapour separating tank. Vapours are removed and condensed whilst the spent shale and oil are passed to separating tanks. T. A. S.

APPARATUS FOR EXTRACTING VOLATILE MATTER.—C. Postel, Assr. to American Shale Reduction Co. U.S.P. 1,458,357 of June 12, 1923. Appl. Jan. 12, 1922.

A retort arranged to rotate about its longitudinal axis is fitted with a jacket containing molten metal. Means are provided for supplying shale to the retort and for withdrawing spent shale and oil vapours. T. A. S.

BITUMINOUS SHALE, OIL SANDS, ETC., PROCESS AND APPARATUS FOR THE RECOVERY OF OIL FROM.—H. Plauson. E.P. 198,705 of June 4, 1923.

Oils are recovered from bituminous materials at temperatures below the boiling point by mechanically removing them with jets of superheated steam. The steam is admitted to the material through nozzles such as are ordinarily used on steam turbines, and moves through the material at a rate of from 300–600 metres per second. The steam is superheated to about 350° C., and after passing through the material falls to 120° C. Largely increased yields of oil are obtained. T. A. S.

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THE PRACTICAL VALUATION OF OIL LANDS.—R. W. Brown. *Oil and Gas J* **22**, (4), 19, 124; (5), 102; (6), 122; (7), 106; (8), 118; (9), 24, 98 100; (10), 126; (11), 106.

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PETROLEUM DIVISION

OF THE

AMERICAN CHEMICAL SOCIETY.

Abstracts of Papers submitted at the Milwaukee Meeting, Sept. 12, 1923.

THE ACCURATE MEASUREMENTS OF HEATS OF VAPORIZATION.—J. H. Mathews, University of Wisconsin, Madison, Wis.

The author has chosen to measure the energy input required to produce evaporation at a fixed temperature rather than to determine the energy freed upon condensation of a vapour. The method requires no knowledge of the specific heat of the liquid, is capable of a high degree of accuracy because of the precision of present-day electrical measurements, and determinations can be carried out more quickly. The author has sought to avoid many sources of error inherent in methods previously used.

The method is suited only for pure liquids having a constant boiling point and operates only at atmospheric pressure. Heats of vaporization of over forty liquids have been determined.

VAPOUR PRESSURES OF PARAFFIN HYDROCARBONS AND HYDROCARBON SOLUTIONS.—E. H. Leslie and A. R. Carr, University of Michigan, Ann Arbor, Mich.

The vapour pressure of n-hexane, n-heptane and n-octane and of various solutions of these hydrocarbons have been measured. Dühring's law has been found to hold for solutions each of whose components exert considerable vapour pressures, as well as for solutions of non-volatile solutes in volatile solvents. The construction of total vapour pressure curves for solutions is thus greatly simplified. Applications of vapour pressure data in the field of fractional distillation are discussed, particularly with reference to the distillation of hydrocarbon mixtures and in the production and utilisation of motor fuels.

THE SPECIFIC HEATS OF LUBRICATING OILS.—E. H. Leslie and J. C. Geniesse, University of Michigan, Ann Arbor, Michigan.

The specific heats of six typical lubricating oils have been measured over a range of temperatures from 100° F. to 290° F. An increase of 35 to 40% in specific heats was found. It is obvious that a change as large as this must be taken into account in engineering work. The variation of specific heats with temperature is not the same for all oils, and it is suggested that further study might disclose some connexion between the temperature rate of change of specific heats and the properties of an oil as a lubricant.

THE EFFECT OF OILINESS ON THE BEHAVIOUR OF JOURNAL BEARINGS. D. P. Barnard, H. M. Myers and H. O. Forrest, Mass. Inst. Technology, Cambridge, Mass.

The writers have made a rather extensive series of friction measurements of a journal revolving in a complete, cylindrical bearing. The object of the work has been to determine the effect of "oiliness" of the lubricant on the position of the transition point from stable, fluid film lubrication to unstable or "adsorbed" film lubrication. The results have shown while oiliness tends to lower the transition point, this effect is too small to be of practical importance. The differences due to oiliness to be expected among commercial petroleum lubricants are negligible.

THE PHYSICAL PROPERTIES OF THE PARAFFIN HYDROCARBONS. Robert E. Wilson and W. Herbert Bahlke, Standard Oil Co., Whiting, Ind.

This paper compiles data from various sources in the literature on the vapour pressures, heats of vaporisation, vapour densities, critical temperatures, etc., for the normal paraffin hydrocarbons. These data are then treated in the light of thermodynamics to choose between discrepant data, to fill in gaps and to extrapolate to higher temperatures and to compounds of higher molecular weight. This results in giving a fairly complete table of approximate values of the more important physical constants for all the hydrocarbons up to tetracosane.

In treating the vapour pressure data especial use is made of the relationship pointed out by Hildebrand. A slight modification of his method of treatment seems to give reliable data over a very wide range in temperature.

THE SOLVENT-INDEX OF REFRACTION METHOD OF DETERMINING OIL IN WAX. Robert E. Wilson and Robert E. Wilkin, Standard Oil Co., Whiting, Ind.

1. This article describes a method developed in the laboratories of the Standard Oil Company (Indiana) for determining the oil content of paraffin wax.

2. The new method consists essentially in dissolving a 5 gram wax sample in ethylene dichloride, cooling to about 0° F., filtering off the wax, washing and evaporating the solvent from the filtrate plus washings. This leaves a residue containing all the oil and the small amount of low melting wax which is soluble even at 0° F. The amount of oil present therein is determined by adding enough of a special solvent to dissolve the residue and measuring the index of refraction, the corresponding oil content being determined by reference to a predetermined curve given in this paper.

3. The method gives good checks on samples containing known amounts of oil. It has been used to show up the magnitude and sources of the errors in the ordinary press method. These tests showed that the error in the press method may readily amount to as much as 2% of oil on samples containing 2 or 3% oil.

NOTE ON AN EXPRESSION OF VOLATILITY FOR GASOLINE. W. A. Gruse, Mellon Institute, Pittsburgh, Pa.

An effort has been made to substitute a more rational term for the 85% point in the arbitrary relation of dewpoint to distillation temperatures of gasolines. It has been shown that by employing a number of

points in the distillation curve a term is obtained which is somewhat more closely proportional to the dewpoint than is the 85% point.

THE TEMPERATURE OF CRITICAL DISSOLUTION WITH ANILINE AS A PHYSICAL CONSTANT OF HYDROCARBONS. Thomas Midgley, Jun., General Motors Chemical Company, Dayton, Ohio.

The temperature of critical dissolution with aniline of hydrocarbons has been advanced as an analytical method for approximate determination of the composition of mixtures of hydrocarbons. The Fuel Section of the General Motors Research Corporation has used the T.C.D. with aniline since 1918 and has not found it of great value in analytical work, but has been found to be a highly desirable physical constant to record, similar in this respect to specific gravity or distillation curve. Taken in conjunction with the specific gravity and distillation curve, it is of material assistance in estimating the probable composition of a sample. In one specific instance it has real value as an analytical determination.

SOME CHARACTERISTICS OF THE HYDROCARBONS PRESENT IN MID-CONTINENT KEROSENE.—C. R. Wagner, Lion Oil Refining Company, El Dorado, Ark.

A closely fractionated kerosene cut from mid-Continent (Oklahoma) crude was separated into two fractions by use of liquid sulphur dioxide. The insoluble portion gave evidence of C_nH_{2n} hydrocarbons, but no hydrocarbons of the paraffin series. The oils soluble in sulphur dioxide from their specific gravity, index of refraction, carbon-hydrogen ratio and boiling points belong to the hydrogenated naphthalene series.

EFFECT OF HIGH-FREQUENCY ELECTRIC DISCHARGES ON THE CRACKING OF PETROLEUM.—J. J. Jakosky, Bureau of Mines, Pittsburgh, Pa.

The effects of both high and low frequency discharges on petroleum hydrocarbons during cracking have been the subject of much discussion in the literature, and patents have been issued in which important commercial utilisation of high-frequency effects is claimed.

Extravagant claims of no carbon formation, polymerisation of uncondensable gas for formation of gasoline, less unsaturation and a more easily refinable product cannot be confirmed by the experiments to date. Whatever effect was obtained by using the high voltage corona (at both high and low frequencies) could be obtained by merely using a higher temperature in the cracking zone.

The results of the work show that little hydrogenation can be obtained by the corona discharge. It seems, further, that commercial application of the corona discharge during cracking of the petroleum will hardly be feasible. Theory relative to ionisation effects in the electric and corona fields is discussed.

A STUDY OF VISCOSITIES OF FUEL OIL MADE FROM PANUCO CRUDE. R. H. Brownlee and W. H. Colburn, Benedum Trees Building, Pittsburgh, Pa.

The paper first describes the development of a new method for making Bunker C fuel oil and fuel oil having lower viscosities from heavy Panuco crude oil. Tables and charts illustrate the laboratory developments which later led to semi-commercial and commercial operation of the process. The general results of these later operations are stated.

It was learned that by stopping distillation at the proper time sufficient

heat treatment of the oil was accomplished so as to produce the required viscosity of fuel oil. While viscosity, flash and fire of this fuel oil could be made to correspond with those of 14-16 gravity fuel oil from light Mexican crude, its gravity was 11-12° Baume.

The behaviour of this fuel oil, upon being stored and handled, is described and discussed.

AN INFORMAL DISCUSSION OF COMMERCIAL ANTI-KNOCK PREPARATIONS, Thomas Midgley, Jun., General Motors Chemical Company, Dayton, Ohio.

Mr. Midgley will discuss the application to commercial use of substances of the type of tetra-ethyl lead.

THE TEMPERATURE ERROR IN SAYBOLT VISCOSIMETERS. W. H. Herschel, Bureau of Standards, Washington, D.C.

In instruments not completely immersed in the bath there is a possibility of temperature error, that is a difference between the nominal and the actual temperature of test. The error in seconds, or in Saybolt viscosity, for a given temperature error, will vary with the viscosity and with the crude of the oil under test. Tests were made at 100° F., with bath at 100.25° F., with an instrument of the U and F type in which the thermometer could be retained in the oil tube during the run, and also with Geo. M. Saybolt instrument No. 108. From a study of data thus obtained, and of the temperature coefficient of viscosity in the outlet tube was lower than the nominal temperature of test, and lower than the temperature in the oil tube at the end of the run.

Abstracts.

Compiled by S. Bowman, A.I.C., P. Dvorkowitz, Ph.D., H. Moore, A.I.C., F.C.S., F. G. P. Remfry, B.A., D.Sc., A.I.C., and T. A. Smith, B.Sc., F.I.C.

General.

New Zealand Mineral Oils.—T. H. Easterfield and N. McClelland. *J. Soc. Chem. Ind.* **42**, 39, 936–938.

Petroleum has been found in many localities, but that occurring at New Plymouth, within twenty miles of an extinct volcano, was the only source likely to produce commercial quantities and that had to be stopped owing to failure of well casings. Analysis of this crude gave :—

Paraffin wax in above	12·0%
Pitch	1·5%

The oil though liquid as it runs from the bore, sets to a semi-solid grease owing to the large content of solid paraffins and was practically free from sulphur.

Further examination of this oil revealed some interesting features, as indicated by the high gravity of the distillates. The benzine and kerosene fractions both burned with a smoky flame, and gave a contraction of 36 and 30 per cent. respectively, on treatment with fuming sulphuric acid, olefines being practically absent. After this treatment these fractions burned without smoking, but the gravity still remained comparatively high, indicating the presence of cyclo paraffins.

Cycloparaffins.

The benzine fraction, after treatment with sulphuric acid to remove the benzenoid constituents, was repeatedly fractionated over a Dufton column, and the following hydrocarbons isolated :—

Fraction.	B.P., °C.	Sp. gr.	Cyclo paraffin.
1 ..	69°– 74° ..	0·7494 ..	Methyl cyclo pentane
2 ..	78°– 83° ..	0·7790 ..	Cyclo hexane
3 ..	100°–104° ..	0·7669 ..	Methyl cyclohexane
4 ..	115°–125° ..	0·7433 ..	Dimethyl cyclohexane + octane
5 ..	146°–152° ..	0·7228 ..	(*n*-nonane)
6 ..	160°–163° ..	0·7884 ..	α-Decanaphthene
7 ..	179°–182° ..	0·7987 ..	Undecanaphthene
8 ..	196°–198° ..	0·8042 ..	Dodecanaphthene

Benzenoid Constituent.

The original benzine, without sulphuric acid treatment, was similarly fractionated and gave :—

Fraction 1.	Boiling below	90°	5·0%
„ 2.	„	between 90°–110°	30·0%
„ 3.	„	„ 110°–140°	47·4%
„ 4.	„	„ 140°–170°	17·6%

On treating fraction 2 with sulphuric acid, and removing the volatile hydrocarbons from the acid layer by evaporation, the benzenoid constituents were regenerated by hydrolysing the sulphonic acids with steam at a temperature above 160° C. These hydrocarbons were fractionated and gave :—

Benzene	B.P. 80°– 81°	9%
Toluene	B.P. 111°	68%
Xylenes	B.P. 120°–145°	17%

Benzene was further identified by conversion to *m*-dinitrobenzene, toluene to 2.4 dinitrotoluene, and the three xylenes by oxidation to the dicarboxylic acids terephthalic acid, isophthalic acid and phthalic acid, respectively.

Toluene and xylene were also found in fraction 3. On similar treatment fraction 4 gave mesitylene and pseudo cumene identified by their trinitro derivatives.

Naphthalene and methylnaphthalenes were isolated from the kerosene fraction boiling between 150°–300° by means of picric acid.

Paraffin Wax.

The wax was repeatedly fractionated under a pressure of 15 m.m., and after recrystallisation from alcohol, melting points and boiling points showed all members of the series to be present from tetracosane to hentriacontane. S. B.

Determination of the Tar Value of Mineral Oils.—E. Casimir. *Petroleum*, 1923, **19**, 763–765.

In the determination of tar value after Holde, by warming with alcoholic solution of caustic potash, acidifying the alcoholic layer, and extracting the residue with benzene, it has been suggested by Marcusson and Schwarz that the treatment with alkali should be followed by treatment with petroleum spirit before acidifying, to avoid the dissolving of some constituents of the oil; and Krammer recommends a further subsequent extraction with benzene. A mineral oil which had undergone the sludging test was found to have a tar value of 1·6 per cent. by Holde's method, 1·4 per cent. by Marcusson and Schwarz's method, and 1·0 per cent. by Krammer's method. The extract with petroleum spirit, on being repeatedly treated with benzene, gave an extract with but feeble acidity compared with that of the primary benzene extract. The amount of the substances extracted by petroleum spirit is greater as the tar value increases. The extract gained by subsequent treatment five times with benzene, after the treatment with petroleum spirit, did not contain all the substances soluble in benzene from the alkaline solution. The insolubility of these substances in petroleum spirit shows them not to belong to the group of hydrocarbons found in mineral oils. While still in solution in the mineral oil the addition of petroleum spirit does not precipitate them. Their acid value is 0·07 per cent. of SO_3, compared with 8·1 per cent. for the substances which occasion the tar value. The method of Holde, Marcusson and Schwarz gave the most accurate results, the method of Krammer leading to the improper exclusion of certain neutral bodies. The addition of water to the acidified alcoholic solution before the first extraction prevents the solution of some of the benzene in the alcohol. Incorrectly high tar values may be obtained, owing to the presence of sulphur dioxide in the sulphuric acid used for drying the oxygen in the sludge test, which results in the formation of tar-forming substances under the conditions of the test. H. M.

PETROLEUM—PREPARATION FROM FATTY OILS. II.—Masakazyn Inouye. *J. Chem. Ind.* (*Japan*), **44**, 78–110.

A complete review of the preparation of petroleum from fatty oils is given. In determining the effect of different catalysers iron, copper, nickel, and brass were used. Carbon adheres to iron so that the latter is not effective. Nickel and brass are only good when fresh. Copper is not as good a catalyser as nickel and brass, but does not lose its efficiency with use. In using a copper catalyst the decomposition should first be carried out in a chamber maintained at 500° C. and the vapours then passed to a chamber maintained at 200–250° C. before condensation. Except for minor differences the same results were obtained from drying, semi-drying, non-drying, animal and sardine oil when the calcium salts of the fatty acids obtained from the oils were distilled. Lead and sodium salts of the fatty acids gave similar results. The gas produced during the distillation is easily combustible, giving 12,000 cal. per litre, and contains 7·4 per cent. CO_2, 7·9 per cent. CO, 33·6 per cent. unsaturated hydrocarbons, 38·2 per cent. saturated hydrocarbons, and 12·9 per cent. hydrogen. T.A.S.

PRESSURE-TEMPERATURE CHART FOR HYDROCARBON VAPOURS.—E. R. COX. *Ind. Eng. Chem.*, 1923, **15**, No. 6, 592–3.

The author has devised a chart by means of which the boiling points of different hydrocarbons over a range of pressures can be instantly read off. He states that "the chart is not presumed to supplant experimentally derived curves, or even to serve as a check, except roughly, upon such data, but is merely offered as a convenient means of readily deriving closely approximate values from a single boiling point where no other data are available." Temperatures from 32 to 700° F., and pressures from 0·01 to 10,000 lb. per sq. in. are given, and the curves being all straight lines makes the chart very easy to read. The idea of the chart was suggested by the Johnson-Duhring relation between the temperature of a saturated vapour and the temperature of steam at the same pressure. The author expresses the hope that the chart may prove of material assistance in solving many problems in petroleum distillation, fractionation and gasoline absorption. F. G. P. R.

DETERMINATION OF WATER IN MINERAL OILS.—L. Losana. *Giorn. Chim. Ind. Applicata.* **4**, 570–3.

By means of agitating mineral oils with sodium amalgam or metallic calcium a rapid and sufficiently accurate determination for technical purposes of the water content can be made. When the moisture content is low the volume of hydrogen evolved can be measured, but in the presence of much water it is easier to calculate from the increased pressure produced within a closed vessel. F. G. P. R.

IMPROVED BITUMINOUS EMULSIONS.—H. A. Mackay. E.P. A, 202,021; B, 202,230; C, 202,231; D, 202,235, of Aug. 9, 1923. (Appl. March 8 and 9, 1923.)

Bituminous material is made into an aqueous emulsion suitable for use as a binding material in road-making or in the coating of wood, stone or felt used for roofing. The bituminous material is melted at about 220° F., and 4 per cent. by weight of oleic acid incorporated by agitation. A solution of caustic soda containing 0·5 per cent. of caustic soda and 30 per cent. of water, calculated on the weight of bituminous material, is slowly added with agitation when emulsification takes place.

On cooling with agitation, no separation of the bitumen takes place, and the resultant emulsion will take up water in any proportions. The fatty acid may be replaced by other fatty acids, or in part by a resin or resin oil. In road-making the mineral aggregate is first moistened with water before adding the bituminous emulsion. Carbonaceous materials are treated with the emulsions in briquet manufacture. T. A. S.

PETROLEUM EMULSIONS—PROCESS FOR TREATING.—W. S. Barnickel, Assr. to W. S. Barnickel and Co. U.S.P. 1,467,831 of Sept. 11, 1923. (Appl. Jan. 4, 1919.)

Roily oil and bottom-settlings are mixed with a sulpho-fatty acid. The impurities and water settle to the bottom of the tank. T. A. S.

BITUMINOUS EMULSION.—C. N. Forrest, Assr. to The Barber Asphalt Co. U.S.P. 1,464,928 of Aug. 14, 1923. (Appl. April 7, 1920.)

A bituminous cement suitable for road material is prepared by emulsifying a bituminous base with an acid mineral oil derivative of sulphuric acid that is freely miscible with water. On drying out the water in road use the cement becomes immiscible. T. A. S.

PITCH CEMENT.—G. A. Henderson and R. G. Erwin. U.S.P. 1,464,480 of Aug. 7, 1923. (Appl. June 16, 1920.)

The cement consists of pitch which has been vulcanised with from one to five per cent. of chlorine and hydrogen sulphide and sulphur dioxide gases in the presence of an accelerator of vulcanisation and protective colloids. T. A. S.

BITUMINOUS CONCRETE.—G. A. Henderson. U.S.P. 1,464,482 of Aug. 7, 1923. (Appl. March 29, 1921.)

Vegetable fibres are impregnated with a bitumen of ductility exceeding 60 cms. at 77° F. Kieselguhr which will pass at 200 mesh sieve, is suspended in the mixture. T. A. S.

BITUMINOUS MASTIC.—G. A. Henderson. U.S.P. 1,464,481 of Aug. 7, 1923. (Appl. Jan. 5, 1921.)

The mastic consists of 43–52 per cent. bitumen, combined with 48–57 per cent. of impalpable mineral dust of exclusively colloidal sizes. It is used to fill the interstices between mineral aggregate in preparing bituminous macadam base to which a bituminous concrete wearing surface of 1½ to 3 inches in thickness is applied. T. A. S.

SEPARATION OF OLEFINES FROM GASEOUS MIXTURES.—B. E. Eldred, Assr. to Carbide and Carbon Chemicals Corporation. U.S.P. 1,465,600 of Aug. 21, 1923. (Appl. Jan. 18, 1918.)

A gaseous mixture containing ethylene, propylene and saturated hydrocarbons is treated to obtain a concentration of the propylene with an absorbent under pressure. The absorbent preferentially absorbs the propylene, and on recovering the gas from the absorbent a higher relative content of propylene is obtained. T. A. S.

APPARATUS FOR PREPARING OIL FOR ELECTRIC TREATMENT.—G. C. Kelley. U.S.P. 1,465,398 of Aug. 21, 1923. (Appl. Aug. 9, 1920.)

Oil from the well is treated to facilitate the separation of water by passing through a tube enclosed in a hot-water drum. A preheater is used to cause the flue gases to heat the oil before it enters the water-jacketed passage. T. A. S.

ROTATING ELECTRODE APPARATUS FOR PETROLEUM EMULSIONS.—H. C. Eddy, Assr. to Petroleum Rectifying Co. U.S.P. 1,466,208 of Aug. 28, 1923. (Appl. Feb. 28, 1921.)

The apparatus consists of a closed tank with means for maintaining a pressure on the contents of the tank. A rotating electrode is centrally disposed in the tank and means are provided for removing the electric potential when the pressure in the tank falls. T. A. S.

THE PRODUCTION OF BENZINE FROM GAS.—J. Strijov. *Grozni Petr. Ind.*, Nos. 5–8, 1923, p. 6–37.

Grozni crude oil and Grozni gas are both very rich in benzine, but the gas is put to no commercial use. The author read a paper before the Technical Association of Grozni in 1902, in which he indicated the necessity for :—

(1) Retaining the gas in the wells.

(2) Taking measures to prevent the escape of gas.

(3) Introducing exploitation of the wells by means of compressors instead of by baling.

(4) Pumping air into petroleum strata in order to increase the productivity of the neighbouring wells.

The chief marketing product of the petroleum industry is benzine, and thus it is necessary to produce the greatest amounts possible from crude oil and gas. There exist four methods of so doing :—

(1) *Distillation.*—The existing plant, both in Grozni and Baku, is very unsatisfactory ; a large percentage of benzine is lost. The fresh Grozni crude oil as received from the wells contains 18 per cent. of benzine, whereas the actual output at present from the refineries is only 6·75 per cent. This loss is chiefly due to the fact that the crude oil is kept in reservoirs which are not hermetically sealed, and to stop this escape of the light products the crude oil should be kept under light pressure.

(2) Producing benzine from gas by means of compressors and absorption.

(3) The production of benzine by "cracking" from kerosene, solar, and other heavy oils.

(4) By increasing the "end" boiling point and including in the benzine fraction Grozni Ligröin, Baku Gasoline, light fractions of kerosene, etc.

The analysis made of the Grozni gas shows the presence of 15–25 per cent. of unsaturated hydrocarbons, and from 50–90 per cent. of methane. According to Strijov's calculations, 17 million cubic feet of gas escape every 24 hours from the Grozni crude. If every 1,000 cubic feet of gas contain four gallons of benzine 28,000 gallons are lost per day. These figures are probably lower than the actual ones. For example, two wells each produce 2,000,000 cubic feet of gas per day. After the benzine has been extracted, the residue should be used in the manufacture of carbon-black.

The water from the Grozni wells contains a considerable quantity of iodine and bromine.

The reserve of crude oil in Grozni is very large, and in the new and old Grozni district the reserves are not less than 250,000 tons. However, the difference between the Grozni and the Baku district is that large areas in the Grozni district are quickly exhausted. They start with big

spouters and gradually diminish. In the course of the last fifteen years, 17 petroleum strata have been exploited and very quickly exhausted. The chief reason for this is that the gas has been allowed to escape freely, and owing to loss of pressure in the wells the crude oil could not be forced up. P. D.

DEPARAFFINISATION.—J. Yelin. *Grozni Petr. Ind.*, Nos. 5–8.

A very difficult technical problem before the suppliers of Grozni Fuel Oil is the removal of the considerable percentage of paraffin scale which is contained in the crude. The fuel oil generally represents a semi-solid mass between a temperature of 15–35° C., and it is evident that the use of such fuel, especially in Russia, where the climate is so severe, represents great complications. A practical solution of this question has not yet been achieved, but very interesting results have been obtained in the course of investigation. Gurevitch's experiments show that the vapour of the fraction which contains most of the paraffin scale is super-heated up to 450° C. on its way to the condenser; thus the transformation of the solid high-melting paraffin scale into low-melting paraffin scale takes place with the separation of a small quantity of gas, and the paraffin scale is obtained in crystalline form, allowing of easy separation by ordinary filter press. Zelinsky is making special investigations of the methods proposed for the deparaffinisation of Grozni crude, and *inter alia* has reported on the application of sulphur dioxide, which effects the deposition of a large percentage of the paraffin scale, and of the asphalt bodies. He found that by these means the temperature of solidification of crude, or of its residue, is lowered by 20° C. P. D.

THE ANALYSIS OF PETROLEUM GAS.—T. Ackermann. *Grozni Petr. Ind.*, Nos. 5–8.

Four samples of gas from two Grozni spouting wells have been analysed as follows: After passing through kerosene, sp. gr. 0·809, 33–65 per cent. are absorbed, thus showing that there is an enormous percentage of benzine in the gas. The temperature at which the gas escapes from the wells is 50–75° C. The respective densities of the gas are: (1) 1·22; (2) 1·35; (3) 1·72; (4) 1·70. P. D.

GEOLOGY OF THE UCHTA OIL-FIELDS.—D. W. Nalivkin. *Y.P.S.T. IV.*, No. 2, p. 235.

The investigation of the groups of Spirifor Anossifi by biometrical methods has shown that S. Anossifi from Central Russia and Taman, and S. Anossifi from the middle devon of the Urals, represent different forms. Further investigations have demonstrated that the beds containing the Spirifer Anossifi of Central Russia and Taman belong to the upper Devon. In this case a section of the Devon of Taman could be represented as follows: (1) the lower part of the section is composed by a suite of shales and sandstones, with diabasic layers; (2) beds with Rhynchonella Meyendorfi; (3) Domanik; (4) Limestone with Spirifer Anossofi; (5) Clays with gypsum; (6) Limestone with Spirifer Archiaci.

The Uchta oil deposit is a regular, very gradually sloping cupola-like anticlinal. The nucleus of cupola consists of a sub-domanikal petroliferous series of beds and by Domanik. The outcrops of Domanik are surrounded by a concentric band of strata containing Spirifer Anossifi. These strata conjointly overlie the Domanik, and are in their turn covered

by clays with gypsum, and beds containing Spirifer Archiaci. Petroliferous area is considerably larger than was previously estimated. The absence of a Graben casts a doubt on the bearing of dislocation phenomena on the origin of the Uchta deposit. P. D.

Distillation and Refining.

Natural Gasoline a Good Refinery Refrigerant.—L. D. Wyant. *Nat. Petr. News.*, 1923, **15**, No. 40, 99–107.

The Bureau of Mines has installed an experimental plant for refrigerating wax distillates by means of highly volatile gasoline in place of the usual ammonia, sulphur dioxide, or carbon dioxide. A fairly detailed working description of the plant is given, which embodies the usual compression and expansion systems, but cooling was done directly by the expanded gases, and not by a brine system. The only disadvantage in the use of gasoline is the risk of fire, and since no corrosion of the plant should occur, the chances of leaks should be negligible, and a well-ventilated building would prevent any dangerous accumulation of gas. A pressure of 120 lbs. appeared all that was necessary when using a gasoline of sp. gr. 0·630, with initial boiling point 15° C., final 119° C., and 50 per cent. over at 58° C. A temperature of −36° C. was obtained in the cooling coil when expanding 1¼ cu. ft. per minute, and a room temperature of 14–16° F. was easily maintained. Tables are given with operating figures of the different runs made, and a chart with the atmospheric, cold room and entering and leaving vapour temperatures. The work described is not intended to be conclusive, but rather to indicate to others the possibility of employing a cheap and readily obtainable cooling medium. F. G. P. R.

Absorption of Gasoline from Natural Gas.—G. A. Burrell. *Chem. and Met. Eng.*, Dec. 29, p. 544–5.

Activated charcoal, *i.e.*, prepared from cocoanut shells, is used for the continuous extraction of gasoline from natural gas. The phenomena of absorption is regarded as taking place in three stages: (1) The formation of a layer of adsorbed gas, one molecule in depth on the surface of the pores; (2) The formation of another layer of varying depth held by the attraction of the solid and vapour; (3) Absorption due to capillary attraction. The first occurs when the partial pressure of the vapour in the gas mixture is practically zero, the second when the first is satisfied and similarly with the third.

Charcoal will retain 10 to 20 per cent. of its weight of gasoline and absorption is selective so that while the gaseous hydrocarbons are at first absorbed they are gradually displaced by the higher members of the series until the absorbent is saturated with these.

The absorbing vessels are arranged in series and when saturation is reached open steam is admitted until distillation is complete. This vessel is now cooled with the residual gas from another vessel, which has the effect of removing the water vapour, thus making it ready for subsequent absorption. S. B.

Desulphurising Mexican Oil Distillates according to the Frash Method.—D. W. Sissingh. *Breun. Chem.* **4**, 113–18 (1923).

Mexican crude oil (sp. gr. 0·976) was distilled according to the method of Engler. When the temperature of the vapour reached 200° C. super-

heated steam was introduced into the still. Five fractions according to sp. gr. were taken: (1) Heavy benzine, 0·820; (2) Kerosene, 0·820–0·850; (3) Gas oil, 0·850–0·900; (4) Spindle oil, 0·900–0·940; (5) Machine oil, 0·940, and over. The original crude oil had a sulphur content of 5 per cent. The fractions had the following sulphur content: (1) 1·27 per cent.; (2) 1·48 per cent.; (3) 2·32 per cent.; (4) 2·6 per cent.; (5) 3·64 per cent., residue 5·7 per cent. Seven desulphurising distillations were carried out with the following desulphurising agents: (1) 500 gr. CuO with 700 g. oil; (2) 1125 gr. CuO, 150 gr. PbO, and 225 gr. Fe_2O_3 with 400 gr. oil; (3) 720 gr. Fe_2O_3 with 600 gr. oil; (4) lux masse (53 per cent. F_2O_3) with oil; (5) a mixture of 1230 gr. CuO, 170 gr. PbO, and 170 gr. Fe_2O_3 with 400 gr. oil; (6) 1600 gr. CuO mixed with oil by use of an air blast, until the temperature at which superheated steam is introduced has been reached (the still was not fitted with an agitator); (7) the same method as (6) with a mixture of 1420 gr. CuO, 180 gr. PbO and 230 gr. Fe_2O_3. Method (6) gave the best results. Fe_2O_3 gave the poorest results. With method (6) the sulphur content of the fractions was (1) 0·78 per cent.; (2) 1·22 per cent.; (3) 1·00 per cent.; (4) 1·28 per cent.; (5) 1·32 per cent. T. A. S.

Fractional Distillation.—E. J. Winter. *Chem. and Met. Eng.*, **29**, 11, 482–4.

The simplest cases of fractionation are those where the constituents are completely miscible in one another, and no chemical affinity exists between them. Mixtures of members of the same series belong to this class. If, however, the constituents have an affinity for each other, or not completely miscible, the problem of fractionation becomes more difficult as the composition of the mixture is constantly changing during its passage through the column. Thus the distillation of alcohol is rendered difficult by the presence of higher alcohols and ethers which are more soluble in ethyl alcohol than in water and ammonia being more soluble in water than alcohol.

The still should have a capacity 50 per cent. larger than the charge, and although the vertical type leaves more room for foaming, the heating arrangement of steam coils is simpler with the horizontal type. The incoming steam should be split up into a number of branches not exceeding 200 diameters. This will condense all the steam at 4 or 5 lbs. pressure at a velocity of 6000 ft. per minute assuming a temperature difference of 30° or 40° C.

Higher velocities increase the rate of heat transmission per degree, but gives a lower temperature difference. When open steam is used the area of perforations should not exceed the cross sectional area of the tube, otherwise the wear is excessive. Columns are usually of the plate or "filler" type, the former being the more efficient, owing to the vapours having to pass through a liquid seal in the form of small bubbles thus exposing large surfaces. The vapours are never in equilibrium with the liquid phase, and have to pass through a misty liquid phase which does not exist in the case of "filler" columns. S. B.

Manufacture of Sodium Hypochlorite.—H. F. Zoller. *Ind. Eng. Chem.*, Aug., 1923.

Efforts to produce a cheap highly-concentrated solution of sodium hypochlorite that would be stable, have so far not met with much success, but if all claims by the present author are substantiated the problem appears to be near solution. The method of manufacture is based on the

hydrogen-ion control system, and the solution is heavily buffered, in order that the reaction will not rapidly change to yield an unstable solution. A solution containing 5 per cent. of available chlorine is considered as a concentrated solution, though solutions with 10 and 15 per cent. can readily be prepared by the same method. Direct chlorination by chlorine gas of a solution containing 10 per cent. of sodium hydroxide, and 2·5 per cent. of sodium carbonate will give a 99–100 per cent. yield of sodium hypochlorite solution containing 5 per cent. of available chlorine with pH approximately 10·5 per cent. It is not necessary to cool the solution during the reaction or to store in a cool place, and temperatures between 15° and 40° C. do not affect the yield. The use of amber bottles is also unnecessary, since it has been found that the influence of daylight on this solution is practically negligible. It is true that increase of temperature accelerates the activity or chlorinating power, but does not affect the internal decomposition at the stable region in pH 10 to 10·5. It has been found desirable to maintain the concentration of sodium carbonate at 2·5 per cent. whatever the amount of sodium hydroxide in the solution to be chlorinated, and in making large quantities it is only necessary to measure the volume of chlorine introduced. With small quantities it is more convenient to follow the reaction by estimating the available chlorine by means of sodium thiosulphate and sodium iodide. Allowing 50 per cent. for labour costs the author calculates the cost of producing 2·5 and 5 per cent. available chlorine sodium hypochlorite at 6·8 and 16·2 cents. per gallon, with soda ash, caustic soda and chlorine at 2, 4 and 7–10 cents. per lb. F. G. P. R.

THE WORKING-UP OF PETROLEUM RESINS.—H. Siebeneck. *Petroleum*, 1923, **19**, 939–941.

After the distillation of the principal products from petroleum, extremely viscous brown red substances come over, which have no appreciable value, and are usually burned under the stills. They contain unsaturated hydrocarbons, and yield cyclic compounds on destructive distillation. This resinous fraction is difficult to free from water owing to its consistency. A similar fraction is produced in the working-up of lignite tar. The fraction from petroleum may be distilled with steam at 400° C., and it amounts to 0·3 per cent. of the petroleum. Its sp. gr. is above 1, its Ubbelohde softening point about 45° C., and its flow point 60° C. The residue was led through a water separator into a steam drying oven. The steam, carrying particles of oil, and the oil vapours were washed and the oil collected. The fraction may be dried in about 12 hours, and the product has the properties of natural asphalt. The yield is 99 per cent. of asphalt, 0·6 per cent. of oil, and 0·4 per cent. loss. The recovered oil has a sp. gr. of 0·950/15, and a viscosity of 3·03 Engler at 50° C. When the fraction was distilled *in vacuo* with steam an oil fraction was again obtained, and 42 per cent. of pure dark-yellow asphalt of excellent quality. The cracking distillation at ordinary temperature with steam gave 31·3 per cent. of paraffin oil, 10·3 per cent. of resin, 47·7 per cent. of coke, and 10·7 per cent. loss. By passing the products of the distillation through a special cracking tube or division, heated by flue gases, 39·3 per cent. of paraffin oil, 51·1 per cent. of coke, and 9·6 per cent. loss were obtained. H. M.

IMPROVEMENTS IN THE PURIFICATION OF OILS.—H. M. Ridge and W. R. Hodgkinson. E.P. 203,354 of March 4, 1922.

Undesirable unsaturated bodies and sulphur compounds giving rise to bad colour or odour in oils are removed by filtering through a porous

medium holding, in a conveniently divided state, an activated or activating material. A convenient porous medium is dehydrated or partially dehydrated bauxite and a suitable activating material is copper or copper oxide. Bauxite in the form of coarse powder is impregnated with a solution of copper acetate or other organic salt of copper, or with an ammoniacal solution of copper and ignited so as to deposit copper oxide, copper suboxide or metallic copper in the conveying medium. The copper should be present to the extent of one per cent. of the mass. Copper oxide, suboxide, or metallic copper may be mixed with bauxite, but does not give so good a result as the impregnation method. After use the filtering medium is freed from organic matter by draining or otherwise and then roasted in an oxidising atmosphere at a temperature not sufficiently high to remove the water of hydration present in the material. The amount of copper used varies from five to twelve per cent. of the weight of oil to be treated according to the nature of the oil.

T. A. S.

DEPHLEGMATORS—IMPROVEMENTS RELATING TO.—D. C. Brandon. E.P. 203,481 of Aug. 1, 1922.

A dephlegmator column is divided in sections, each section being traversed by a bundle of cooling tubes. Vapours rising through the dephlegmator are partially condensed on the cooling tubes. Steam is fed into the bottom of the dephlegmator to assist in the revaporisation of the condensate. Fractions are taken from the various sections of the dephlegmator and by controlling the steam and cooling water to the apparatus cuts can be obtained that do not require redistillation. Arrangements are provided for continuously removing the water condensing in each section.

T. A. S.

DISTILLATION OF PETROLEUM—IMPROVEMENTS RELATING TO.—M. Simonoff, M. Beninson and F. Gros. E.P. 200,118 of Jan. 4, 1922.

Distillation is carried out by means of electrically heated elements distributed uniformly about the liquid. The elements consist of chrome-nickel wire or ribbon in the form of spirals or grills. More economical use of heat is claimed, and stills may be built of lighter material than when ordinary firing is used.

T. A. S.

PURIFICATION OF MINERAL OILS.—A. E. Dunstan. E.P. 201,223 of July 23, 1923. (Appl. April 22, 1922.)

Mineral oils, particularly naphtha are treated with chlorine in the presence of water, or with an aqueous solution of hypochlorous acid. A naphtha containing 0·146 per cent. of sulphur, when acted upon by 0·426 per cent. chlorine in the form of chlorine water gave, on distillation, after treatment with weak alkali, a distillate of 0·720 sp. gr., and having a sulphur and chlorine content of 0·01 per cent. The distillate does not attack copper, has an excellent smell and gives no reaction for sulphur. The residue from the distillation on filtration through absorptive substances gave an oil of good colour and smell, and containing 0·04 per cent. of sulphur.

T. A. S.

PURIFICATION OF HYDROCARBONS.—A. G. für Chem. Ind. E.P. 193,802 of Aug. 7, 1923. (Convention, Germany, Feb. 21, 1922.)

The hydrocarbon to be purified is treated with liquid sulphur-dioxide, the operation being made more economical by giving the hydrocarbon

repeated extractions with sulphur dioxide, partly saturated with the resinifying bodies it is desired to remove and a final extraction with pure sulphur dioxide. The process is applicable to the treatment of lignite tar oil, to the purification of commercial anthracene or montan wax. T. A. S.

OIL STILL.—P. Heffler, Assr. to H. C. Simmons. U.S.P. 1,463,698 of July 31 1923. (Appl. Jan. 28, 1922.)

A setting for an oil still is fitted with two sets of chequer-brick work. The products of combustion are made to pass through each set of chequer brickwork in turn, the other set having a current of air passed through it. T. A. S.

APPARATUS FOR TREATING HYDROCARBONS.—B. Andrews. U.S.P. 1,464,918. Aug. 14, 1923. (Appl. Sept. 13, 1919.)

The apparatus consists of a tower having a centrally disposed liquid elevating conduit having its lower end terminating in a body of liquid in the base of the tower. The vapour to be treated is forced into the lower portion of the conduit, whereby liquid and vapour are raised up the tower, the liquid falling back in a finely-divided state, and being brought in intimate contact with the vapour. T. A. S.

REGENERATION OF ANHYDROUS ALUMINIUM CHLORIDE.—G. R. Stewart, Assr. to A. S. Hirsing and W. C. Hollister. U.S.P. 1,464,373 of Aug. 7, 1923. (Appl. Nov. 26, 1921.)

Aluminium chloride is separated from petroleum coke by oxidising the carbon content to the gaseous state, thus freeing the occluded aluminium chloride which is converted into aluminium oxide and chlorine, and by further heating in a reducing atmosphere aluminium chloride is regenerated. T. A. S.

GAS SCRUBBER.—H. G. Johnston. U.S.P. 1,465,397 of Aug. 21, 1923. (Appl. Sept. 13, 1922.)

Gas is scrubbed free from oil or the like, by passing it along with water, down a conduit centrally disposed in a packed tower. The bottom outlet of the conduit is kept covered with water, oil and tar collecting on the surface of the water being continually removed. Gas on escaping from the bottom of the conduit ascends the packed tower, the packing being kept moistened by means of water-sprays. T. A. S.

MOTOR FUELS.

MOTOR ALCOHOL—ITS THEORY AND USE.—J. P. Foster. *Sugar News*, 1923, 2, 521.

The best motor fuel is considered to be a mixture of alcohol and ether, but the corrosive action of the products of combustion of this fuel is a serious problem. The author has shown that the best means of neutralising this corrosion is the addition of primary, secondary, or tertiary amines to the fuel previous to use. F. G. P. R.

BENZOL AS A MOTOR FUEL.—A. C. Fieldner and G. W. Jones. *Chem. and Met. Eng.*, Dec., **29**, p. 543.

The crude benzol or light oil as obtained in coke-oven practice contains corrosive sulphur compounds in addition to unsaturated bodies, which oxidise and polymerise to gummy deposits. Comparative tests were carried out to note the performance of this class of fuel before and after refining. The engine used for the purpose was a four-cylinder Lycoming with 3½ ins. bore and 5 ins. stroke, coupled directly to an electric dynamometer and revolution counter. The speed of the engine was kept constant, and an orifice plate inserted between the carburettor and manifold, in order to keep the amount of air-fuel mixture constant. The air-fuel ratio was adjusted to 10·1 to 1 and 13·5 to 1 from ultimate analysis of fuel and composition of exhaust gases. A testing period of 40 hours at 1000 r.p.m. was adopted, cooling water kept at 150° F., and readings taken every 15 mins., and samples of exhaust gas every hour. At the end of the run the fuel consumption was noted. Also the weight of carbon and any gummy deposits found on the cylinder, piston, valves, etc. Under these conditions the crude was found to give heavy deposits of gummy matter, which caused sticking valves after only 16 hours run, with both air-fuel ratios. Acid refined and Gel refined benzol both ran the full test of forty hours without serious trouble, and showed only small amounts of gummy deposits. S. B.

FUEL FOR INTERNAL-COMBUSTION ENGINES.—A. G. Bloxam. (Comm. from J. D. Riedel, A.G.) E.P. 202,805 of Aug. 30, 1923. (Appl. July 6, 1922.)

A mixture of 50 pts. by weight of tetrahydronaphthalene and 50 pts. of petrol of sp. gr. 0·725, or a mixture of 50 pts. tetrahydronaphthalene, and 40 pts. heavy petrol of sp. gr. 0·760, and 10 pts. gasoline of sp. gr. 0·650, provides a fuel of sp. gr. about 0·850, and which, owing to its low ignition point, may be used in any normal motor car better than ordinary petrol. The speed of explosion of such a mixture is slower than that of pure petrol, and hence "knocking" in high compression engines is avoided. The mixture may be further mixed with other fuels such as benzene. Instead of tetrahydronaphthalene, decahydronaphthalene may be used. T. A. S.

MOTOR FUEL.—F. W. Rohrs. U.S.P. 1,460,767 of March 7, 1923. (Appl. Dec. 17, 1921.)

The motor fuel consists of a mixture of 1 to 3 parts acetone, 25–35 pts. petroleum distillate, and 50–80 pts. benzol. The proportion of benzol increases between the limits set with increase in temperature. T. A. S.

CRACKING.

CRACKING OF PETROLEUM OIL IN GAS-LIQUID PHASE.—G. Egloff. *Ind. Eng. Chem.*, 1923, **15**, No. 6, 580–3.

In the United States at present 28 per cent. of the gasoline output is produced by cracking, and henceforward few refineries will be able to run at a profit without a cracking plant capable of dealing with heavy oils. In 1921, out of 443 million barrels of crude oil, 88 million barrels of straight run, and 34 million barrels of cracked gasoline were produced.

By 1931 it is estimated that a total of 300 million barrels of gasoline will be required, of which half will be produced by cracking. To be commercially successful a cracking plant must be able to operate on kerosene, gas oil, fuel oils, topped crudes and crude oil depending on the prevailing market price. The vapour phase processes can only treat light oil distillates of the kerosene character, and their high cost per gallon, small throughput, and large gas losses, make them now of doubtful commercial utility. The catalytic method, such as that employing aluminium chloride, is also ruled out by the cost of the process, though the products leave nothing to be desired. In the liquid-gas phase process, of the Burton type, the dangers due to explosions of the stills and the large fire risk, militate against their use, whilst only light oils can be operated on. The gas-liquid phase process, however, greatly diminishes the fire and explosion risk, owing to the small amount of oil present in the fire zone, and the great strength of the pressure reaction vessels. Moreover, heavier oils, such as fuel oils and topped crude can be used in these plants. The author describes the well-known Dubbs' plant which is operating successfully in the United States. F. G. P. R.

HYDROGENATION OF PARAFFIN BY THE BERGIUS METHOD.—H. I. Waterman and N. J. Perquin. *Proc. Acad. Sci. Amsterdam* **26**, 226–30 (1923).

The cracking of paraffin in a 2500 c.c. autoclave, gas heated to 435° C., alone and in the presence of hydrogen at pressures varying during a particular experiment from 40 to 110–20 atm., was studied. The paraffin had the following properties: $d\frac{15}{15}$ 0·913, Shukoff solidifying point 50·6° C. and McIlkiney bromine No. (addition) 0·5. Transparent yellow to red oils were obtained in the Bergius experiments, but in the absence of hydrogen dark opaque oils were obtained. Small negligible quantities of carbon were deposited in both cases, and complete conversion to oil and gas occurred. In both methods the distillate to 220° C. was 22·8–25 per cent. after one hour at 435° C., 36–42 per cent. after 2 hours, 52–56 per cent. after 3 hours, and 57–60 per cent. after 4 hours. The gas loss in every case was below 7 per cent. Products of lower specific gravity were always obtained in the hydrogen treatment experiments than in the simple cracking operation. The amount of hydrogen absorbed is considerable, and slightly lower bromine addition numbers of the products (19, 20·8 and 20·8, as against 27·6, 26·1 and 23·9). The Bergius method is considered by the authors to be superior to cracking without hydrogen under pressure. T. A. S.

LIGHT OILS AND GASOLINE—METHOD AND APPARATUS FOR MANUFACTURE OF.—The Texas Co. (Communicated to A. E. White.) E.P. 200,534 of Jan. 12, 1922.

The apparatus consists of a tube system in which oil is heated to cracking temperature and cracking chambers, in which cracking takes place. The operation is carried out at pressures of 100–400 lb. per sq. in., according to the oil used, and the character of the product desired. The oil to be cracked is passed through the tube system at such a rate that the temperature at which incipient cracking takes place is only attained as the oil leaves the tube and enters the cracking chamber. Auxiliary heat, if desired, may be applied to the cracking chamber, this most conveniently being affected by insulating only the upper part of the cracking chamber, and applying heat to the lower portion, or, if desired, the chamber may be heated within electrically. With the type of oils employed in cracking operations the temperature of the cracking chamber

is maintained at 750–850° F. (399–454° C.), and within the coil a temperature of 800–950° F. (426–510° C.). The vapour from the cracking chamber is submitted to fractional condensation, the lighter fractions being removed and the heavier condensate being returned to the process, where it is used to preheat the incoming oil whilst the heavy non-volatile portions containing carbon are taken from the bottom of the cracking chamber. A plurality of connected, cracking chambers may be used in conjunction with one heating coil. A scraping mechanism is provided to remove carbon deposited on the walls of the cracking chamber. Gauges are provided for indicating the height of the oil in the cracking chambers which also contain vapour. T. A. S.

Hydrocarbon Oils—Cracking of.—A. F. M. Seigle. E.P. 190,723 of Dec. 20, 1922.

Petroleum, peat, shale, resin, naphthalene or lignite oils and heavy tars are fed through a preheater to the lower part of a vertical annular retort, heated by a flame projected from an oil burner down the central cavity. The vapours pass over discs on which are placed chips of copper, iron, aluminium, nickel, etc., to act as catalysts. The vapours are heated to a temperature of 550–650° C., and pass from the retort to coolers, which are surrounded by jackets containing calcium chloride solution or water, which by liberation of steam under pressure are maintained at constant temperature, *e.g.*, 250–300° C. and 180° C., respectively. Uncondensed vapours are passed to a water-cooled condenser, and either to a compressor or scrubber, in which they are washed with heavy oil.
T. A. S.

Cracking Hydrocarbon Oils.—W. F. Garagher and W. A. Gruse. E.P. 174,885. (Additional to E.P. 174,087 and 174,088.)

In high-pressure stills employed for the cracking distillation of petroleum oils, etc., the formation of carbon deposits on the still bottoms is prevented by means of circulating the oil. Eduction tubes are fitted in the still, and by means of a small quantity of gas (*e.g.*, steam, inert gas or gases produced by the cracking) led into the bottom of the tubes, a circulation of oil is brought about on the air-lift principle. F. G. P. R.

Method and Apparatus for Dehydrating, Distilling and Cracking Hydrocarbon Oils.—E. O. Linton. E.P. 180,657 of May 3, 1922. (Convention (U.S.) May 26, 1921.)

Heavy oils such as Mexican-Panuco and other asphaltic oils or residuals, are treated to remove water, distilled and the vapours cracked. The dehydrator still is situated above the distillation still, oil being carried from the bottom of the dehydrator to the lower portion of the cracking still by means of a vertical pipe. Oil entering the dehydrator flows in thin films on to the hot walls, the water being removed as steam. It then flows to the bottom of the cracking still, where it is vapourised, the vapours being brought to cracking temperature in the upper portion of the still. After cracking the vapours are removed, condensed and redistilled. Asphaltic residues are taken from the cracking still.
T. A. S.

The Continuous Dehydration and Distillation of Tar or Oils, and the Cracking of Residuals.—T. D. Wilton and Wilton's Patent Furnace Co. E.P. 200,933 of July 24, 1923. (Appl. April 24, 1922.)

The patent is an addition to E.P. 127,700 (*J.*, 1919, 529A). In this

patent tar or oil is passed through a series of heating coils under pressure, and suddenly released into an expansion chamber after each coil, whereby the lighter constituents are volatilised. The present patent is extended to include the cracking operation which is carried out in a coil heated to 350° C. or upwards, instead of 230–300° C. as in the former patent. To facilitate replacement of the coil heated to the higher temperature the coils are placed side by side instead of concentrically. T. A. S.

DISTILLATION AND CRACKING OF MINERAL OILS.—N. V. S. Knibbs. U.S.P. 1,459,156 of Oct. 5, 1920.

Heavy hydrocarbon oils to be cracked or distilled are sprayed into the top of a chamber, where they meet a current of hot gas, consisting of products of combustion of heavy residue oils, entering at the bottom of the chamber. The temperature of the entering gases may be from 600° C. to 1200° C., whilst at the top of the chamber the temperature may be from 200° to 400° C. Atmospheric pressure may be employed, or pressures ranging from 50 to 200 lb. per sq. in. F. G. P. R.

CRACKING OF HYDROCARBON OILS.—F. T. Manley, Assr. to the Texas Co. U.S.P. 1,462,143 of July 17, 1923. (Appl. Oct. 10, 1917.)

Oil is charged into an elongated vertical still and maintained at a temperature and pressure sufficient for cracking. A desired level is maintained in the still, cracked products being removed through a condenser, and some residuum being also removed. The heavier portions of the distillate are returned to the bottom of the still for further cracking. T. A. S.

HYDROCARBONS—PROCESS OF TREATING.—L. H. Manning. U.S.P. 1,462,068–9 of July 17, 1923. (Appl. April 16, 1921.)

Oils are cracked by heating them in a closed chamber, means being provided for agitating the oil. The heating chambers are tubular, and are traversed by rods carrying inclined vanes. On rotating the rods the desired movement is conveyed to the oil. Cracking is carried out at a temperature of 700° F. T. A. S.

TREATMENT OF HYDROCARBONS.—W. F. Rittman. U.S.P. 1,462,247 of July 17, 1923. (Appl. Feb. 5, 1917.)

Oil is cracked in two stages. In the first stage the operation is carried out under such pressure that the oil remains in the liquid phase. After this stage the desired product is removed and the residue vapourised and cracked under pressure in the vapour phase. T. A. S.

MANUFACTURE OF GASOLINE.—R. Yates. U.S.P. 1,465,752 of Aug. 21, 1923. (Appl. July 6, 1920.)

Oil is cracked in both gaseous and liquid phases in cylinders fitted with an inner rotating drum. Vapour is forced into the drum, causing oil previously present in the drum to occupy the annular space between the drum and the cylinder. The exterior of the cylinder is heated, the oil being partially cracked and heat being conveyed to the vapour in the drum, which also undergoes decomposition. By reducing the pressure low boiling products are removed, and are subjected to fractional condensation. T. A. S.

Lubricating Oils and Waxes.

A Melting-point Bridge for Petrolatums, Shale Oils and Shale-oil Distillates.—L. C. Karrick and V. F. Parry. *Ind. Eng. Chem.*, 1923, **15**, No. 6, 600–2.

Owing to the difficulty of even approximately determining the melting point of greases and such like substances by the usual methods the authors have devised an apparatus in which a rod is electrically heated in such a way that any required constant temperature difference exists between the two ends, whereby a constant and even fall of temperature is maintained along the length of the rod. A thin line of grease is spread along the rod, and a movable air blast blowing on to the line of grease is started at the hot end and moved at a fixed rate towards the cooler end. The melted portion of the grease is obliterated by the blast which, however, leaves the unmelted portion undisturbed. This point is quite sharp, and by means of a formula employing the temperatures at each end of the rod, and the distance of the unmelted point from the hot end the melting point of the grease can be calculated. F. G. P. R.

Vapour Pressures of Solutions of Benzene, Hexane and Cyclo Hexane in Various Types of Lubricating Oils, with Molecular Weight Data.—R. E. Wilson and E. P. Wylde. *Ind. Eng. Chem.* **15**, 801–9 (1923).

Oils prepared from California, asphalt base and paraffin base petroleum, and castor oil were used. For a given mean molecular weight the deviations from Raoult's law arranged the oils in the following order of increasing tendency to absorb the volatile hydrocarbons—California, asphalt base, paraffin base and castor. This order is the order of increasing molecular weight for a given viscosity. Graphs and tables are given, showing the behaviour of the mineral oils and volatile solvents over a considerable range of temperature. T. A. S.

Purification of Hydrocarbons.—Whitehall Petroleum Co. (Communicated from A. Greenspan. E.P. 203,098 of June 22, 1922.)

The present methods of dewaxing oil distillates, consisting of chilling and filtering or centrifuging the distillates, presupposes that all the wax is crystalline if the process is to be successful. In the presence of amorphous wax neither process is successful. Both types of wax may be removed if the oil is diluted with kerosene, chilled so that the crystalline wax is deposited and separated by filtration. The filtrate is then distilled till it is free from amorphous wax, when satisfactory lubricating oil stock remains. T. A. S.

Lubricating Liquid to be Added to Motor Spirit.—T. Aspden and W. J. Tolley. E.P. 202,830 of Aug. 30, 1923. (Appl. Aug. 11 1922.)

The mixture consists of 50 per cent. mineral co'za oil, of sp. gr. 0·850, 30 per cent. mineral oil of sp. gr. 0·908, 10 per cent. of sperm oil of sp. gr. 0·895, 7·5 per cent. naphthalene, 1·5 per cent. camphor gum, and 1 per cent. of oil soluble dye. The addition of 1 liquid ounce of the composition

to two gallons of petrol or benzol is sufficient to satisfactorily lubricate the cylinder walls, piston rings, and valve guides of a small engine, and will prevent the deposition of carbon. T. A. S.

SEPARATION OF WAX FROM MINERAL OIL STOCK.—C. H. Hapgood, Assr. to The DeLaval Co. U.S.P. 1,464,707 of Aug. 14, 1923. (Appl. Oct. 22, 1921.)

The wax containing lubricating oil stock is submitted to two successive refrigerations, whereby the wax is thrown out of solution and submitted to centrifugal force. The first refrigeration is produced by passing the wax free oil through a heat exchanger, where it cools the incoming stock. The second refrigeration is produced by cooling with ammonia.
T. A. S.

SHALE, COAL AND ASPHALT.

MEIHUIZEN'S METHOD OF DRYING APPLIED TO SOLID FUELS.—D. J. W. Kreulen. *Chem. Weekblad*, 1923, **20**, 529–532.

The method indicates the content of water indirectly by the loss sustained on heating. The space between two concentric vertical copper pipes, each closed at the bottom and open at the top, is occupied in the lower part by a liquid, the boiling point of which represents the temperature at which drying is to be conducted, while the upper part, filled with small stones, acts as a condenser. The pipes are surrounded by a double mantle to prevent air cooling. A copper tube is welded to the bottom of the inner copper pipe, and ends about 1 cm. above the surface of concentrated sulphuric acid in a narrow-necked 10-litre flask. A frame carrying metal pans, in which the substance to be dried is placed, is inserted in the inner copper tube, and at the top of this a thermometer indicates the temperature. Heat is applied to the bottom of the outer copper pipe, the inner pipe acts as a chimney, and air is drawn through the apparatus from the flask. Samples of coal were dried, using water, xylol, and dichlorbenzol as liquids, and concordant results were obtained in a shorter time than by the usual method. The results being plotted in the form of curves, a correction for oxidation could be introduced. The method is simpler than the American system, as it does not necessitate the use of compressed air. The best results were got by drying 5 minutes at 175° C. H. M.

VERTICAL RETORT FOR THE DISTILLATION OF SHALE AND BITUMINOUS MATERIALS.—Rasen Schaefer and Co. E.P. 190,109 of July 27, 1923. (Appl. May 1, 1922. Convention (Germany) Dec. 7, 1921.)

The distillation chamber is formed between two heated walls enclosing a relatively narrow space. This space is divided by vertical venetian-blind like gratings into several compartments. Two spaces on either side of a pair of gratings are filled with the material to be distilled, the space between the gratings being used for the escape of the products of distillation. The gratings may be fitted so as to be easily removed as a whole, and in cases where distillation is facilitated by the use of steam this is introduced into the space normally used for the reception of vapours so that it is driven through the gratings across the material to be distilled. T. A. S.

RETORT FOR OIL SHALE.—H. L. Brown. E.P. 202,107 of Aug. 16, 1923. (Appl. June 14, 1922.)

Shale in lump form is fed into an oblique rotating retort, through which it progresses in a shallow layer. The material is subjected to light physical shocks as it proceeds through the retort, whereby it is broken up and fresh surfaces presented for the disengagement of gases. Steam is admitted during distillation. The retort works continuously, the lighter vapours being removed at the inlet end, and the heavier vapours at the exit end. T. A. S.

HYDROCARBON OILS FROM SHALE—PROCESS AND APPARATUS FOR EXTRACTION OF.—D. T. Day. U.S.P. 1,467,757 of Sept. 11, 1923. (Appl. July 12, 1919, and 1,467,758, Appl. Aug. 22, 1919.)

Shale is fed into an insulated chamber from whence it passes to an externally-heated horizontal retort. The vapours produced in the retort pass through the mass of shale in the insulated chamber, whereby part of the organic content of the shale is removed. In U.S.P. 1,467,758 the above apparatus and method is used to crack heavy hydrocarbon oils, which are previously mixed with shale to form a dry non-caking mixture, which is fed to the retort. The shale is utilised as a scouring medium to keep the interior of the retort free from carbon. T. A. S.

HEAT TREATMENT OF OIL SHALES.—E. Stansfield. U.S.P. 1,459,932 of April 3, 1920.

A retort for the treatment of oil shales, coal, peat, etc., has a bed plate composed of carborundum slabs inclined at an angle greater than the angle of repose of the material, which passes over the heated surface in narrow streams. The temperature of the bed plate increases towards the lower end. The thin layers of material require less time for complete removal of volatile constituents, which are drawn off at several points along the length of the retort in order to prevent undue cracking by contact with the heated surface. F. G. P. R.

OIL SHALE RETORT.—W. M. Strong. U.S.P. 1,462,023 of July 17, 1923. (Appl. July 20, 1920.)

Shale is caused to traverse a long distillation chamber by means of a mechanical conveyor. The distillation chamber is heated from below by furnace gases. Ducts convey furnace gases over the openings provided for charging and discharging shale, thus preventing the admission of air and the oxidation of the hydrocarbons. T. A. S.

OIL SHALE RETORT.—J. Mayes and R. Homrighous. U.S.P. 1,465,277 of Aug. 21, 1923. (Appl. Aug. 16, 1920.)

Shale is caused to traverse a horizontal retort the under surface of which consists of an enclosed bath containing molten lead. Arrangements are provided for removing vapours at several points along the length of the retort. T. A. S.

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Abstracts.

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General.

Measurement of the Relative Absorption Efficiencies of Gas Absorbent Oils.—R. E. Wilson and H. S. Davis. *Ind. and Eng. Chem.* **15**, 9, pp. 947–950.

Although the methods employed by the authors were similar in that they measured the vapour pressure of the absorbed material in the absorbent, the work was carried out independently. Seven of the absorbent oils used were petroleum distillates and, in addition, one coal tar distillate. The former were represented by :—

1. Distillate boiling between 230° and 360° C.
2 and 3. The lower and higher boiling fractions of 1.
4 and 5. Being 2 and 3 after acid treatment.
6 and 7. Cracked distillates have boiling ranges of 2 and 3.

In view of the importance of the recovery of "light oil" from coal gas, benzene was used as a means of determining the absorption capacities of these oils. Accurate molecular weight determinations were made in order to ascertain whether the absorption capacity varied inversely with these (Raoult's law). Incidentally, it was noticed that the molecular weights of the higher boiling fractions were about 40 points higher than the low boiling fractions, and for the same boiling range there was little difference between the straight run and the cracked distillates. Also, that acid treatment reduced molecular weights 8 to 10 points. In each case the author used a standard solution of benzene in the absorbent and measured the vapour pressure, but whereas the percentage of benzene and temperatures used differed, the results, calculated from percentages of saturation pressures at a given temperature, plotted against the molecular fraction of benzene in solution, were in close agreement. In both sets of results, oils 2, 4 and 6 (low-boiling fractions showed a slight deviation above Raoult's law, and oils 3, 5 and 7, a slight deviation below, No. 1 being intermediate. These results showed that the volume of benzene absorbed by the different petroleum distillates under given conditions is substantially the same for all. The coal tar oil is a rather better absorbent, but the disadvantages such as cost, high viscosity and difficulty of handling rule out its use. S. B.

Bath Temperatures of Viscometers of the Saybolt Type.—W. H. Herschel. *Ind. and Eng. Chem.* **15**, 9, pp. 945–6.

Instruments of the Saybolt and Barbey type, where the oil containing tube projects above the level of the bath, are liable to give appreciable differences between the nominal temperature of the test and the actual temperature of the oil during the run. With the Saybolt viscometer the usual directions are that the bath must be held constant at such a temperature as will maintain the desired temperature in the standard oil

tube, but as the thermometer is usually withdrawn from this tube at the commencement of flow, the variation in temperature of the oil during a run is unknown and must depend on several factors. This variation renders it difficult, if not impossible, to get agreement, and the authors have so modified the instrument as to have this under control, and then be able to apply the necessary correction factors. The chief dimensions of the instrument are retained, but the several factors have been worked out in order to give reproducibility and conversion to terms of absolute viscosity. S. B.

CENTRIFUGING PETROLEUM-REFINERY EMULSIONS.—E. E. Ayres. *Trans. Amer. Inst. Min. Met. Eng.*, No. 1276–P, Oct., 1923.

The function of centrifugal force and gravity is to bring the suspended globules into contact and cause their coalescence by rupturing the films around the globules. Centrifugal force has little more effect than gravity in causing coalescence, but is much better for subsidence, which is proportional to the force applied. The adjustment of the centrifuge rotor depends on the extent of coalescence that is desired. One reason for this is that the hydrostatic pressure of emulsions is not proportionate to their specific gravity, for instance, 0·96 in. of water may be required to balance 1 in. of emulsion having a sp. gr. of 0·86. In the case of a water-in-oil emulsion, the water globules move outwards, and the coalesced water layer represents dead space, reducing the capacity of the rotor for the oil phase. Similarly the oil globules in an oil-in-water emulsion move inwards, and the oil layer represents dead space. Where industrial requirements allow of a partial separation, for example, the presence of 1% of oil in the water, and 1% of water in the oil, greatly increased yields may be obtained, say, in the proportion of ten to one. When "cut production" is dehydrated in the field the moisture is reduced to 1–2%, and the oil stored, when the emulsion accumulates in the tanks, particularly if reagents have been used in the field. When the percentage of inorganic matter in the emulsion is high, it may be agitated with a solution of caustic soda, soda ash or sodium silicate, heated and settled, and afterwards centrifuged. Water-soluble colloids tend to break water-in-oil emulsions, the most efficient being sodium-rosin soap containing 60% dry matter. When crudes contain salt water, removing the water is tantamount to removing the salt. Some low viscosity crudes, containing under 2% of water, can be reduced to 0·1% moisture by centrifuging at high capacities. To the more viscous crudes sodium-rosin soap may be added, and the same result attained by centrifuging. If a lubricating distillate and sulphuric acid are passed through a centrifuge, acid sludge and sour oil free from suspended sludge are discharged, but the sour oil deposits acid sludge on standing. The oil will have received efficient acid treatment. The viscosity of the oil prevents the entire separation of the acid sludge. Well-coalesced sludge may be separated from even viscous oils by centrifuging. The emulsions formed when water is agitated with sour oils are water-in-oil, and require an hydrophile colloid for their resolution. A sour oil may be washed efficiently by agitating with starch solution, and afterwards neutralised with a caustic soda solution. 0·1% by weight of starch has been found to be sufficient. When sour oils are neutralised with caustic soda the soap formed tends to stabilise the water-in-oil emulsion. The addition of water causes the emulsion to break down. Solutions of starch or other hydrophile colloids do not aid the separation of alkaline emulsions. They may be resolved by agitation with 20–60% of water and centrifuging. H. M.

INDUSTRIAL ADSORPTION OF GAS AND VAPOUR WITH ACTIVATED CARBON.—A. B. Ray. *Chem. Met. Eng.*, 1923, **29**, No. 9, 354–9.

Activated carbon is now largely used in industrial and military gas masks and smoke helmets, for extracting gasoline from natural gas, benzol and light oils from coke oven gases, vapours of volatile solvents from the air and for many other purposes. For industrial application the carbon must have mechanical strength, and relative high apparent density. Pure gas or vapour is held by carbon in two ways: (*a*) rather loosely by capillary condensation, and (*b*) more firmly by the specific attractive forces of the carbon. The total amount held is called the "saturation" value, and the more firmly held part (*b*) the "retentivity" or specific adsorptive capacity. When the concentration of gases or vapours is low, retentivity is the chief factor in evaluating an adsorbent, but when the concentration is high the total sorptive capacity is important. Various types of activated carbon are compared with the different forms of inorganic porous bodies—such as silica gel and sil-o-gel—in their capacities for adsorbing a variety of gases and vapours. In all cases it is shown that carbon, when activated in the proper way, is superior to the inorganic materials owing to "the specific attraction—*i.e.*, the force subsisting between the solid and the gas molecules—being much greater with charcoal than with silica." As a rule, gases and vapours are sorbed in the order of their boiling points, but with activated coconut charcoal the more chemically reactive substances are preferentially adsorbed. Thus silica gels adsorb water vapour in preference to hydrocarbon vapours, whilst charcoal does the reverse, and for this reason proves greatly superior in practical working. Carbon that is saturated with water will, in fact, adsorb hydrocarbon vapours, which actually displace the water in the carbon. This, however, only occurs at ordinary temperatures, and recovery of vapours held by the carbon can be readily effected by passing steam at 100–150° C. through the carbon, which can then be rapidly cooled by sluicing in cold water, and is ready for use without drying, an important advantage over other adsorbent materials which have to be dried before use. Vacuum may be applied to assist in removing the adsorbed vapours and the carbon may be externally heated by steam coils or the like where water soluble products are being recovered, though even with acetone direct steam can be employed as a large proportion of the former can be condensed almost free from moisture. The more important fields in which activated carbon can be advantageously used are: (1) separation and recovery of vapours and gases; (2) purification of gases; (3) storage of gases; (4) catalysis; and (5) production of high vacua. Examples are given of all these uses, with a lengthy list of references to technical and patent literature bearing on the subject.

F. G. P. R.

NEW ZEALAND MINERAL OILS.—T. H. Easterfield and N. McClelland. *Chem. and Ind.*, 1923, **42**, 936–8.

Three classes of oil kauri, shale and natural mineral oil have been produced in the country. Kauri oil is obtained by the distillation of peat containing kauri gum, and is a complex mixture like crude rosin oil. Oil shales are found in large deposits in the Southland district, whilst the only considerable amount of natural mineral oil occurs at New Plymouth. This oil is practically free from sulphur and basic constituents, but contains 12–13% of paraffin wax and all fractions have a specific gravity somewhat higher than similar fractions from other mineral oils. The following constituents have been identified—methylcyclopentane,

cyclohexane, methylcyclohexane, α-decanaphthene, undecanaphthene. dodecanaphthene, benzol, toluol, three xylols, naphthalene, α-methyl, naphthalene, 1 : 4-dimethylnaphthalene and the paraffins from $C_{24}H_{50}$ to C_{31}, H_{64}, excluding C_{27} H_{56}. F. G. P. R.

SEPARATION OF GAS MIXTURES BY DIFFUSION INTO STEAM UNDER PRESSURE.—F. Fischer, H. Schrader, and A. Jaeger. *Brennstoff.-Chem.*, 1923, **4**, 289–296.

By passing gas mixtures and steam in counter current on either side of a porous earthenware wall, and thence through condensers to receivers a substantial separation of the light and heavy constituents of the gas mixture can be achieved. Medium gas and steam velocity and relatively low steam pressure give the best results. The earthenware wall may be satisfactorily replaced by Raschig rings, but sand and narrow tubes do not give such good results. Illuminating gas containing 44–45% of hydrogen gave 70–74% and 95% of hydrogen in the first and second runnings through the apparatus. F. G. P. R.

PAINTING OF OIL TANKS.—*Chem. Met. Eng.*, 1923, **29**, No. 18, 792.

The U.S. Bureau of Mines are carrying out large scale tests of the value of different coloured paints for reducing evaporation losses from oil tanks. Previous tests indicated that evaporation from tanks painted white averaged 1–1½%, less than from those painted red, and 2½% less than black tanks. Dark colours absorb heat and highly glossy surfaces are less absorptive than mat surfaces. Tin plating and aluminium paint gave the best results, but iron coated with tin rapidly corrodes and aluminium paint rapidly flakes off. In a particular case the rise in temperature of the benzine in a tank painted black was 140% greater than in a similar white tank. F. G. P. R.

WATER SOLUBLE SULPHONIC ACIDS FROM PETROLEUM OILS.—H. E. Humphreys, F. M. Rogers, and O. E. Bransky, Assrs. to Standard Oil Co. of Indiana. U.S.P. 1,474,933 of Nov. 20, 1923. (Appl. Oct. 25, 1918.)

Water soluble sulphonic acids are obtained by treating petroleum oil with strong or fuming sulphuric acid and separating the acid sludge. The sludge is then washed with hydrocarbon oil, diluted with water, heated and allowed to settle. The sulphuric acid layer is drawn off and the sulphonic acids purified. The acids have a combining weight of about 360. T. A. S.

APPARATUS FOR SEPARATING OIL FROM OIL-BEARING SANDS AND ROCKS.—W. J. Reilly. U.S.P. 1,475,028 of Nov. 20, 1923. (Appl. Feb. 17, 1922.)

The apparatus consists of a still fitted with a heating element and a grinding device situated near the top of the still. The still contains hot oil, into which the oil-bearing material is introduced. Two separators attached to the bottom of the still serve for the removal of unvolatilised material. T. A. S.

SHORT CUTS IN TESTING NATURAL GAS BY CHARCOAL METHOD.—R. C. Alden. *Nat. Petr. News*, 1923, **15**, No. 48, 89.

The charcoal method of testing natural gas is now generally accepted. for the purpose of settling royalties and evaluating new fields. In making the test the specific gravity of the gas must be known in order that proper saturation of the charcoal may be effected. The specific gravity gives the approximate and the charcoal test the precise yield of gasoline obtainable from the gas. In order to facilitate testing a calcu-

lator on the slide rule scheme has been devised by the author. The specific gravity of the gas to be tested being unknown, it is possible by the use of the calculator to at once read off the number of cubic feet of gas that have to be passed through the charcoal in order to get complete saturation, the approximate number of gallons of gasoline contained in 1000 cu. ft. of the gas, and the rate at which the gas should be passed through the charcoal tester. F. G. P. R.

Uses and Limitation of Charcoal Process.—G. C. Oberfell and R. C. Alden. *Refiner*, 1923, **2**, No. 11, 7–10.

Activated charcoal has now become a practical means of recovering condensible constituents from natural gas and other vapours. In general a cycle of three containers is employed, in one of which the charcoal is adsorbing, in the second the already saturated charcoal is being steamed out to remove the condensed liquids, whilst in the third the steamed charcoal is being cooled by the stripped gases issuing from the first container. The charcoal at present used for gasoline extraction is almost exclusively made by the dry distillation of cocoanut shells, with a subsequent treatment with steam at 850° C. to completely remove all volatile organic matter. The supply of shells is quite equal to the demand in spite of fears expressed on that point, but manufacturing costs still remain high, as only 12% of the raw material is converted into suitable charcoal. Treatment of the dry distilled shells with selenium oxychloride in place of steam as an activating agent has been successfully performed, but the charcoal is stated to be not so good. Direct comparison between charcoal extraction and other processes is very difficult, on account of the varying conditions of use and the meagre details available for comparison. In all actual trials the charcoal has extracted from 25 to 50% more gasoline from natural gases than oil adsorptive processes, but it should be stated that the latter could be improved to bring them more in line with charcoal if it were economic to do so. Charcoal is also stated to produce a more stable product than oil plants. The best method of comparison was to test the gas before extraction at different oil adsorption and charcoal plants by means of the regulation charcoal adsorption tester, and obtain the average ratio between the actual plant figures and charcoal tester figures. In this way both processes were compared with one standard method. As the mean of very many tests the charcoal plants were found to exceed the charcoal tester figures by 55% more than the oil extraction plants, but this figure is considered to be excessive. So far as installation costs are concerned there is not much difference, though the running costs of an oil adsorption plant decreases more rapidly with increase of size of plant than in the case of charcoal plants. As, however, charcoal can work profitably on much leaner gas then oil extraction plants, the best methods for using the former are: (*a*) on lean gases; (*b*) on the stripped gas issuing from oil extractors; (*c*) as portable plants in exploiting new gas fields. By working in this way large losses at present taking place could be avoided, and further profits secured. F. G. P. R.

Evils of Crankcase Oil Dilution in Internal-Combustion Engines.—W. F. Parish. *Nat. Petr. News*, 1923, **15**, No. 42, 45–8.

Lubricating oil has three functions to perform: (1) to keep the bearing surfaces apart, and so avoid wear; (2) to maintain a lubricating seal between the piston rings and cylinder walls to preserve compression; and (3) to burn cleanly without leaving a deposit of carbon. Before the year

1913 the usual lubricating oil had a viscosity of 180–190 Saybolt seconds at 100° F., which would now be classed as extra light motor oils, and would not be used. In those days, however, very volatile fuels were obtainable, and no trouble was experienced from dilution of the lubricating oil by heavy ends from the fuel with the resultant wear on bearings and moving parts. Owing to increased demand for motor spirit this was gradually increased in specific gravity and final boiling point, carburettors and engines being improved to accommodate the heavier fuel. These improvements, however, did not extend to prevention of heavy unburnt fuel passing through to the crankcase, and the author shows the effect of this by the increase in renewals of moving parts of the engine that has progressively occurred since 1913. Crankcase dilution increases regularly to a point where a balance occurs and excess leakages of fuel are thrown off as vapour through the breather. At this point, however, the lubricating oil has been reduced to the consistency of a heavy kerosene, and has lost most of its properties as a lubricant. A chart is given, showing that extra heavy oils will bear a dilution of 16%, medium oils, 8%, and light oils only 2% before the viscosity falls below the danger point of 180 Saybolt seconds at 100° F. Charts are also given which show that, as the end boiling point of commercial fuels was raised, so the demand for heavier lubricating oils increased, but at the same time the sales of spare engine parts went up in far greater proportion. The author recommends more frequent draining of the crankcase as being much cheaper than replacement of worn out parts, and insists that on no account should the crankcase and engine be washed out with anything but clean lubricating oil. The original paper should be consulted for statistical details brought forward to prove that an increased use of lubricating oil is an economy for the motorist. F. G. P. R.

Distillation and Refining.

Refining of Petroleum with Liquid Sulphur Dioxide.—L. Edeleanu. *Z. angew. Chem.*, 1923, **36**, 573–580.

While a distillate from a Pennsylvanian crude oil gives an illuminating oil of excellent quality when refined by the usual process, distillates from Roumanian and some other crudes yields illuminating oil of very poor quality, which generates smoke and an unpleasant odour in burning, and has a poor lighting value. This is the case with crudes containing a high proportion of aromatic and unsaturated heavy hydrocarbons of high carbon content, even when the physical properties of the distillate correspond with those of a good illuminating oil, as the behaviour of the oil as an illuminant depends upon its chemical constitution. It was found possible to burn such oils satisfactorily in lamps of a special construction, but this condition of use diminished their commercial value. The unsaturated constituents of such oils could be isolated by conversion into sulphonic acids and treatment with superheated steam. The same oils when deprived of their contents of aromatic and unsaturated hydrocarbons were found to burn quite satisfactorily. The separation of heavy hydrocarbons by treatment with sulphuric acid was found to be impracticable commercially for Roumanian oils, as the loss on this operation was 25%. The separation of the hydrocarbons by organic solvents, such as alcohol, acetic acid and various ethers, was too costly for adoption. On shaking up a distillate from Bustenari crude, of sp. gr. 0·82, with liquid sulphur dioxide and allowing the mixture to stand, two layers were formed, the under layer consisting of sulphur

dioxide, and the hydrocarbons soluble in that compound. The upper layer was a purified distillate of sp. gr. 0·803, and the under layer, or extract, had a sp. gr. of 0·865. The distillation limits of the two portions of the oil were approximately the same. The paraffin hydrocarbons are difficultly soluble in liquid suphur dioxide, and almost totally insoluble when the temperature is below —10° C. The naphthenic hydrocarbons are difficultly soluble at low temperatures, but their solubility increases with rise in temperature more rapidly than that of the paraffins, while the aromatic and unsaturated hydrocarbons of the illuminating oil fraction are soluble in all proportions at low and high temperatures. On adding to a petroleum distillate in a closed vessel liquid sulphur dioxide a certain amount of the latter dissolves in the oil till the saturation point is passed, when further additions of sulphur dioxide form a layer containing dissolved aromatic hydrocarbons. In treating a distillate containing a high proportion of aromatic hydrocarbons, it is necessary to keep the temperature down at —10° C., as at —7° C., or above, the two separate layers are not formed, and much of the paraffin hydrocarbons dissolve. The amount of sulphur dioxide required is about two-thirds of the weight of the distillate. By treatment of a Bustenari illuminating oil fraction with sulphur dioxide its lighting power was increased from 7 to 14 Hefner candles. The distillation of the extract gives a solvent, known as white oil or turpentine substitute, and a residue which finds use as fuel oil for Diesel engines. This treatment has been applied to the separation of the hydrocarbons in lignite and shale oils, from the light fractions of which benzol and toluol are obtained, and from the lubricating oil fractions, by separation of aromatic and resinous substances, lubricating oils of good cold test. During the sulphur dioxide treatment of petroleum distillates their sulphur content is considerably reduced. The principal parts of the apparatus for the Edeleanu process are two coolers, in which the distillate and the liquid sulphur dioxide are cooled to the necessary temperature, the mixer, in which the mixing of the two liquids and separation into layers takes place, and two evaporators, in which the refined product and the extract are freed from sulphur dioxide, which is recovered, recondensed, and cooled for further use. Diagrams of the plant for intermittent and continuous treatment are given. The process serves for the production of valuable illuminating oils from low-grade crudes, rich in asphalt and sulphur, and the production of lubricating oils of low cold test from petroleum and from lignite tar oils. H. M.

Treatment of Liquid Hydrocarbons.—A. E. Dunstan. E.P. 204,078 of April 22, 1922.

Aliphatic liquid hydrocarbons containing unsaturated bodies, *e.g.*, cracked spirit, are refined by treatment with chlorine water or hypochlorous acid, and subsequent hydrolysis of the chlorine compounds formed with aqueous alkali. The large losses which occur when such spirit is treated with sulphuric acid are avoided and a motor fuel of high detonation value is obtained. T. A. S.

Purification of Cracked Spirit.—F. G. P. Remfry. E.P. 205,808 of July 22, 1922.

Cracked hydrocarbons are treated in the vapour phase with dense porous activated charcoal, whereby the compounds which cause "gumming" are polymerised. The operation is carried out at a temperature 10°–20° C. above the final boiling point of the liquid. Cracked spirit of final boiling point 200° C. was passed at the rate of 2·5 c.c. per minute over 85 grms. of activated charcoal maintained at a temperature of 220° C.,

and the vapours condensed. Sweet smelling colourless distillate (400 c.c.m.) was obtained, which did not go off colour or deposit gum on keeping. The treatment may be carried out in the presence of hydrogen, or under pressure, but this is not essential. After use the charcoal can be reactivated in the usual manner. T. A. S.

MOTOR SPIRITS AND PETROL—IMPROVEMENTS RELATING TO PRODUCTION OF.—F. Tinker. E.P. 205,289 of Sept. 5, 1922.

For the satisfactory recovery of petrol on a "topping" plant it is essential to have a cooled dephlegmator column, and also apparatus for the recovery of the petrol carried by the permanent gases. In the present invention both effects are obtained, without expensive plant, by the use of cooled condensate from the fractionating column to scrub the permanent gas. This oil is then returned to the top of the fractionating column, where it assists in the work of dephlegmation by cooling the vapours, and at the same time gives up the petrol it has removed from the permanent gas. T. A. S.

IMPROVEMENTS IN THE PURIFICATION OF OILS, WAXES, AND THE LIKE.—Oil Refining Improvements Co., and J. J. Hood. E.P. 206,535 of May 4, 1922.

The removal of impurities from oil by materials such as bauxite is a function of the relative dielectric constants of the oil, and the impurity as well as the nature of the bauxite. A bauxite may be so active that instead of working only on the impurities it removes other substances and allows the impurities to pass through the bauxite filter. The bauxite may be degraded by the addition of iron impurity in the form of iron sulphate, caustic soda, salts of nickel, or copper, or alkali salts, before roasting so that only the desired impurities are removed from oil when the latter is filtered through the bauxite. The amount of degrading necessary for a bauxite to be used on a given oil is determined by experiment. The facts explain many of the anomalies observed when working with bauxite. T. A. S.

CASING-HEAD GAS—PROCESS AND APPARATUS FOR CONDENSING.—G. E. Gray. U.S.P. 1,470,116 of Oct. 9, 1923. (Appl. Oct. 28, 1920.)

The casing-head gas is passed through a column fitted with cooled layers of finely-divided metallic contact material, the layers being continuously cooled by circulating cooling liquid through them. The condensed liquid is drained away and the uncondensed gases are led to a second similar apparatus, where, in addition to being cooled, they are also scrubbed with liquid hydrocarbon. T. A. S.

SEPARATION OF HYDROCARBONS FROM WATER.—W. A. Brown. U.S.P. (A) 1,472,384 and (B) 1,472,385 of Oct. 30, 1923. (Appl. Feb. 24, 1919.)

(A) Water is separated from oil by mixing the emulsion with alcohol, and distilling under pressure, so that alcohol and light hydrocarbons volatilise but not the water. (B) Mixtures of hydrocarbons and water are mixed with finely divided absorbent clay, and the mixture heated. On allowing to stand the water and clay are precipitated. T. A. S.

DISTILLATION OF HYDROCARBON OILS—IMPROVEMENTS IN.—T. E. Robertson. Communicated from Power Specialty Co. U.S.A. E.P. 206,029 of Nov. 23, 1922.

Hydrocarbon oils are heated in a tube or other still to a temperature sufficiently high to volatilise all the fractions it is desired to distil. The

oil is then passed to a separator, from whence the vapours are passed to condensers and the residue to a heat interchanger, where it is used to preheat the incoming crude oil. The condensers are in series and consist of vertical chambers traversed by vertical tubes through which air is pumped. Each condenser is controlled so that it removes a required fraction from the vapours. The hot condensates are also used for preheating oil, and the hot air pumped through the condenser tubes is used in combustion under the still. T. A. S.

Refining of Oil.—C. B. Forward. U.S.P. 1,473,402 of Nov. 6, 1923. (Appl. Jan. 9, 1919.)

Oil is passed through a coil suspended in the flue of a steam boiler, coking of the oil being avoided. It is then passed to vessels where it is treated with the steam from the boiler and fractionated. T. A. S.

Petroleum Refining Apparatus.—C. B. Watson. U.S.P. 1,474,395 of Nov. 20, 1923. (Appl. April 2, 1920.)

A horizontal cylindrical still is fitted with a dome and is partially filled with molten metal maintained at a predetermined level. The molten metal is carried to the top of the dome and forced downwards through a nozzle, into the centre of which petroleum oil is delivered. A good contact between the molten metal and the oil is ensured, and vapours formed are carried off to a condenser. T. A. S.

Resolution of Emulsions—Process and Apparatus for.—C. H. Hapgood, Assr. to De Laval Separator Co. U.S.P. 1,474,670 of Nov. 20, 1923. (Appl. March 25, 1922.)

A reagent, adapted to cause the separation of the constituents, is added to a flowing stream of emulsion which is then treated in a centrifugal machine. The back pressure of the flowing stream is used to regulate the rate of supply of the emulsion and the treating agent. T. A. S.

Process for Treating Oils.—F. W. Bayard, Assrs. to E. J. du Pont de Nemours Co. U.S.P. 1,474,136 of Nov. 13, 1923. (Appl. Aug. 17, 1918.)

Olefines and other unsaturated bodies are removed from tar distillate, containing chiefly benzol, toluol, and solvent naphtha by treating the distillate with 70–75 per cent. sulphuric acid and removing the acid sludge. The loss of aromatic bodies is avoided. T. A. S.

Motor Fuels.

Weighted Index Number for Motor Fuels.—W. A. Gruse. *Chem. Met. Eng.*, 1923, **29**, No. 22, 970–1.

Wilson and Barnard have pointed out that the dew point of gasolines bear an approximate linear relationship to the temperature figure at which 85% of the spirit has distilled off during the conventional Engler distillation test (*J. Ind. Eng. Chem.*, 1921, **13**, 906, and *J. Soc. Automotive Eng.*, 1923, **12**, 287). This 85% point bears a better relationship to the dew point than any other point on the distillation curve, but owing to the possibility of producing by suitable blending gasolines that have the same 85% point, but entirely different characteristics, the author conceived the idea of obtaining a better approximation to the dew point by means of utilising several points on the distillation curve. The points chosen were selected on account of their importance in actual use in motor engines, and were the temperatures at which 10, 20, 50, 70 and 90%

distilled off, and also the final boiling point. The latter is important, owing to the fact that a small amount of heavy ends in a gasoline raises the dew point markedly and hence decreases the effective volatility of the fuel. The temperature figures at the above points are added together and give the index figure for the gasoline under examination, the lower the index figure the better the fuel. It was found that by plotting the dew points as determined directly by experiment against the index numbers obtained by the above method a straight line curve was obtained when working on a large variety of motor fuels. The index number thus obtained is therefore an equal indication with the dew point of the value and properties of any motor fuel, and it is claimed gives a much better differentiation between gasolines than does the ordinary distillation curve. Dew points being difficult to determine can therefore be superseded by index numbers, which can be obtained during ordinary routine testing, and as the former are known to place motor spirits in their order of merit as found in actual practice the latter are equally as informative, but far more readily obtainable by the ordinary worker. F. G. P. R.

LIQUID FUEL—UTILISATION OF ALCOHOL IN THE PRODUCTION OF.—A. E. Dunstan and F. B. Thole. E.P. 205,367 of Nov. 16, 1922.

The necessity of using a substance as a 'binder' to render stable mixtures of liquid hydrocarbon and alcohol is avoided by cooling mixtures of hydrocarbon and alcohol so that a denser layer separates out. This layer, consisting mainly of aqueous alcohol and mineral oil is removed, and the residual mixture is found to be stable; *i.e.*, not to separate into two layers, at ordinary temperatures. A mixture consisting of 10% commercial alcohol (containing about 5% water) and 90% petrol gives a denser layer, at 20° C., having a volume of 5–6% of the mixture, whilst the upper layer consists of 94–95% petrol, about 6% of alcohol and a very small amount of water. On further cooling to −10° C. a further dense layer of about 0·6 to 0·7 per cent. of the mixture is obtained, containing about 75% petrol, 25% alcohol, and a very small proportion of water. The upper layer consists of 5–6% alcohol, 94–95% petrol, and a water content too small for measurement. This mixture is homogeneous and stable and is satisfactory for use as fuel. T. A. S.

LIQUID FUELS—IMPROVEMENTS RELATING TO.—P. Lennox and J. C. Calder. E.P. 205,582 of July 21, 1922.

A cheap motor fuel is prepared, consisting of 46% alcohol, 50% water, and 4% of hydrocarbon. The alcohol used may be denatured, additional oxygen may be obtained by adding ·67 gr. barium peroxide, and ·2 gr. hydrochloric acid per litre. The fuel may be further improved by the addition of ·67 gr. picric acid per litre. T. A. S.

GAS DETERMINATION SAVES MOTOR FUEL.—*Chem. Met. Eng.*, 1923, **29**, No. 14, 620.

Experiments by the U.S. Bureau of Mines have proved that substantial saving of fuel can be obtained by adjustment of the carburettors on motor cars based on analyses of the exhaust gases. An increased mileage, with a saving of 22% of fuel, was obtained with a fleet of government motor lorries. A portable carbon dioxide indicator removes all guesswork in adjusting the carburettor, and is a feasible proposition in practice.
F. G. P. R.

MOTOR FUEL.—W. C. Perry. U.S.P. 1,471,916 of Oct. 23, 1923. (Appl. Feb. 27, 1923.)

The fuel consists of a solution of naphthylamine in petrol. T. A. S.

MOTOR FUEL.—J. J. Murphy. U.S.P. 1,471,566 of Oct. 23, 1923. (Appl. July 5, 1922.)

The fuel consists of 40 parts coal oil, 40 parts benzol, 5 parts ether, and 20 parts alcohol. T. A. S.

LIQUID MOTOR FUEL.—A. A. Backhaus, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,474,135 of Nov. 13, 1923. (Appl. May 28, 1921.)

The fuel consists of petroleum distillate and butyl alcohol of over 98% strength. T. A. S.

CRACKING.

CATALYTIC CRACKING OF HEAVY HYDROCARBONS.—Erdöl u. Kohle-Verwertung A.-G. E.P. 204,458 of Aug. 1, 1922.

Oil is cracked by heating to a suitable temperature (350-380° C. in the case of paraffin wax) in a still containing a layer of catalyst, *e.g.*, activated charcoal. Contact of the oil with the catalyst is ensured by pumping the oil from the bottom of the still and returning it at the top above the layer of catalyst. Light hydrocarbons are removed as vapour through a dephlegmator. From wax (mp. 51° C.) a 50–80% yield of light distillate sp. gr. ·755 is obtained and the residue in the still consists of viscous oily matter and a solid of high setting point. The deposition of carbon is avoided. T. A. S.

VAPOUR-PHASE CRACKING PROCESS.—L. de Flores. *Refiner and Nat. Gasoline Mfr.* **8**, 7–10 (1923).

A vapour phase cracking equipment consists of 8 furnaces, each containing a vertical coil of 1·5 in. tubing reduced to 1 in. in the cracking zone. Oil is passed through these tubes at a velocity of 16,000 ft. per minute, and thence to an expansion chamber. The oil is pumped through strainers at 200 lb. pressure, and reduced by needle valves before entering the tubes to 75–125 lb pressure. The pump and tube pressures are recorded on the same dial, thus readily indicating the condition of the tubes. The lining of the expansion chamber can be readily removed for cleaning. A plant dealing with 1000 barrels a day costs 25c. per barrel, and a further 15c. for refining. T. A. S.

LOW-BOILING HYDROCARBONS—PROCESS OF MANUFACTURE OF.—G. L. Prichard and H. Henderson, Assrs. to Gulf Refining Co. U.S.P. 1,473,316 of Nov. 6, 1923. (Appl. Nov. 19, 1921.)

A mixture of high-boiling oil and aluminium chloride is maintained at a high temperature, and the low-boiling vapours produced are removed. Additional high-boiling oil heated to a higher temperature than the mixture and maintained substantially in the liquid phase is continuously added to replace the removed cracked material. T. A. S.

DISTILLING AND CRACKING PETROLEUM.—J. L. Gray. U.S.P. 1,474,147 of Nov. 13, 1923. (Appl. Oct. 25, 1918.)

Petroleum is injected into the bottom of a series of tubes filled with molten material. The tubes are connected at the top to a common header by which the treated petroleum is removed. T. A. S.

METHOD OF TREATING PETROLEUM.—D. W. Hoge, Assr. to Bash Oil and Refining Co. U.S.P. 1,474,755–6 of Nov. 20, 1923. (Appl. (1) June 24, 1916; (2) May 25, 1918.)

Heavy petroleum oils are cracked by passing them in the vapour phase through a long narrow conduit maintained at cracking temperature. The conduit consists of a number of parallel pipes connected at the ends so that the vapours frequently and abruptly change direction of flow. The formation of carbon is thus prevented. In (2) the cracking coil is suspended in a bath of molten lead maintained at a temperature of 650–1000° F. T. A. S.

APPARATUS FOR TREATING OIL.—G. Egloff and H. P. Bonner, Assrs. to Universal Oil Products Co. U.S.P. 1,470,353 of Oct. 9, 1923. (Appl. Aug. 23, 1920.)

Oil is cracked by heating in a coil connected with a vapour chamber. Vapours from the chamber pass to dephlegmators, which are connected to a common header from whence they pass to a condenser. Liquid condensing in the dephlegmator is returned to the coil. T. A. S.

LUBRICATING OILS AND WAXES.

CENTRIFUGAL REMOVAL OF WAX FROM PETROLEUM LUBRICATING OILS.—L. D. Jones. *Trans. Amer. Inst. Min. Met. Eng.*, No. 1275–P, Oct., 1923.

In the "cold settling" process formerly employed for dewaxing steam-refined cylinder stock (600° F. fire test) the cylinder stock was mixed with about 1½ times its volume of naphtha, decoloured by treatment with fuller's earth, heated to dissolve all the oil and wax, and slowly chilled to about 15° F. The precipitated wax settled out by gravity, and the supernatant liquid was drawn off and the naphtha distilled off, leaving a still residue of bright stock. The residue was worked up for petrolatum. The yield of bright stock, of a cold test of 50° F., was seldom above 70%, and the settling process was irregular and uncertain. Attempts to separate the wax in cream separators were unsuccessful. The wax separated in centrifuges differs from the slushy liquid separated by gravity, and being a plastic did not act as a liquid in the rotor. A rotor of the hydrostatic balance type gave a discharge of wax only for short periods with difficulty. The difficulty was overcome by the invention of the so-called "solid discharge." A stratum of a third liquid substance, heavier than the substances to be separated, was maintained within the rotor. Calcium-chloride brine at the temperature of the chilled oil was used as a carrier liquid. An improvement has been made by supplying the carrier liquid in a jet, directed within a ring on the rotor, this being known as the "top feed." By this means refrigeration of the carrier liquid is rendered unnecessary, the wax may be melted on its discharge, and mixture of the carrier liquid with the chilled oil is avoided. The oil mixture is heated to 100° F. before cooling, until all wax is dissolved, and then cooled at the rate of about 3° F. per hour. Oil chilled by this method to –10° F. will yield a bright stock of 15° F. cold test. Sudden, or "shock chilling" precipitates the wax in a colloidal state, when it cannot be separated. The dilution of the stock must be sufficient to provide for free suspension of the precipitated wax. The presence of asphalt and colouring matter lowers the cold test of an oil, therefore the stock is refined to the required colour before dewaxing. A yield of 85% of 15° F. cold test bright stock may be obtained. The process is

applicable to any stocks containing wax in an amorphous form, and especially to "long residuum"; a steam-refined still residue amounting to 30% of the crude oil. In this way it is possible to obtain 10% of 200-sec. viscosity neutrals of cold test below 25° F., and 15% of bright stock of cold test below 15° F. from a Pennsylvanian crude oil.

H. M.

LUBRICATION OF HEAVY OIL ENGINES.—H. Moore. *Oil Engineering and Finance* **2**, 525.

High viscosity oils are unnecessary on Diesel engines. Oil for lubrication of enclosed high-speed engines must have high flash (over 420° C.) to avoid crank-case explosions. Lubrication of the compressors is more difficult than that of the engine. Acetic and formic acids, in addition to water, have been found to be present in the liquid from compressors. The formation of these acids is minimised by the use of oils of low iodine value, but these oils do not give such good film maintenance as asphaltic oils. The formation of acids is less in 3 stage than in 2 stage compressors. The lubrication of engines not employing compressed air is simpler than the lubrication of those using compressors. Compounded oils containing 5–7½% of rape, cocoanut, or lard oil are suitable for the cylinder lubrication of these engines, as are asphaltic base oils. Hot bulb engines require oil of higher viscosity.

T. A. S.

"GERM PROCESS" LUBRICANTS.—H. M. Wells. *Oil News* H., 11, 16, 17–18 (1923).

The addition of 1% fatty acids to mineral oils increases the lubricating value of the oil by 17%. The grooving of bearings and journals, when properly carried out, also increases the efficiency by 17–22%. Emulsification in steam and turbines has also been overcome by the addition of fatty acids to the lubricants.

T. A. S.

SWEATING OF PARAFFIN WAX—IMPROVED APPARATUS FOR.—F. Ealing and S. H. Johnson and Sons. E.P. 205,245 of July 26, 1922.

A wax-sweating apparatus is built up after the manner of a filter-press, and consists of a number of chambers in which cakes of wax are placed, the spaces between the chambers being heated. The apparatus is built up of interchangeable members.

T. A. S.

WAX-FILTER.—H. F. Glair and E. J. Wonnacott, Assrs. to Standard Oil Co. of Indiana. U.S.P. 1,474,899 of Nov. 20, 1923. (Appl. Aug. 11, 1920.)

A filter press is fitted with heating elements, so that after wax has been deposited from chilled oil, on the filtering medium it may be removed by melting.

T. A. S.

NON-SLUDGING OILS—PROCESS OF MANUFACTURE OF.—H. R. Moody. U.S.P. 1,472,882 of Nov. 6, 1923. (Appl. Feb. 6, 1918.)

Oil is made non-sludging by treating it with nascent hydrogen at a temperature so low that cracking does not take place. The sludge-forming constituents are reduced and rendered non-sludge-forming.

T. A. S.

USED OIL—PROCESS FOR RECLAIMING.—J. C. Miller. U.S.P. 1,469,426 of Oct. 2, 1923. (Appl. Jan. 7, 1921.)

Waste lubricating oil from gasoline engines is recovered by heating and injection of steam to remove volatile substances. Soap solution is then

added, and more steam injected, whereby dirt and foreign matter are coagulated. Heating is continued until all water is expelled, and the solids are then separated from the oil. T. A. S.

Shale, Coal and Asphalt.

Factors Controlling Behaviour of Asphalt Mixtures.—J. C. Morrell and L. Kirschbraun. *Chem. Met. Eng.*, 1923, **29**, No. 9, 362–4.

In evaluating the usefulness of asphalts for industrial purposes a knowledge of such properties as penetration, melting point, viscosity, ductility, cementing value and elongation are necessary. The author describes these properties in mixtures of steam refined and air-blown asphalts in order to indicate the possibilities of the study of such systems from a physicochemical viewpoint. The phase-rule, $f = n-p+2$, where f indicates the degrees of freedom, *i.e.*, temperature, pressure and concentration; n, the number of components and p the number of phases, as used in the case of alloys, is considered with a view to its applicability to mixtures of asphalts. Pressure and the vapour phase may be ruled out of the variables to be considered, and therefore the formula is reduced to $f^1 = n-p+1$. Two mixtures of air blown and steam refined asphalts were studied, and curves are given of the six properties enumerated above, tracing their values between 0 and 100% of each constituent. These results are discussed in relation to the above phase-rule formula, and the hope is expressed that a systematic study along the lines of solid and liquid hydrocarbon mixtures, which as yet has hardly been touched, will be undertaken by others. The above method of study was also applied to liquid mixtures, the two components being a fuel oil and the residuum obtained by cracking the same. No break was found in the viscosity curves, but a point of economic importance was noticed in the cold tests where it was found that, although the set point of the original oil was only lowered 18° F. when the proportion of the mixture was 70% fuel oil, to 30% cracked residuum, yet the set point fell 50° F. when the mixture was in the proportion of 60% to 40%. F. G. P. R.

Research Work on Oil Shale.—*Chem. Met. Eng.*, 1923, **29**, No. 22, 971.

A description is given of the work that is being carried on under the U.S. Department of the Interior in Colorado, Utah and Indiana. Two objects are kept in view: (*a*) to determine the conditions of retorting that produce the highest yields of the best oils from various shales; and (*b*) a thorough study of methods of refining shale oils. At the Boulder laboratory, Colorado, an experimental vertical retort, a large horizontal retort and a small assay retort are being tried. The effects of time and temperature of preliminary heating, holding at various temperatures, use of steam in the retort, etc., are being experimented with. The horizontal retort gives a lower yield of poorer oil than the others. The Bureau of Mines has concentrated on refining gasoline from shale oil, and has obtained good products. Assay retort studies of many shales will prove how far foreign practice should be adhered to with American shales. At Salt Lake City the problems being attacked are the distribution of sulphur and nitrogen in oil shale and its products, the separation and examination of kerogen, the approximate and ultimate analyses of shale and ash, determination of best conditions for recovering ammonia or other nitrogen products and the thermal decomposition of oil yielding materials from oil shale. F. G. P. R.

OXIDATION PHENOMENA IN THE DETERMINATION OF VOLATILE MATTER IN COAL AND ANTHRACITE BY THE MODIFIED BOCHUMER METHOD, AND THE INFLUENCE OF ASH CONTENT ON THE RESULT.—D. J. W. Kreulen. *Chem. Weekblad*, 1923, **20**, 553–555.

A series of experiments was made on pure carbon, Welsh anthracite and cannel coal, dried for 4 hours at 85° C., and tested by heating for 10, 15, 30, 45 and 60 minutes. The results plotted gave straight-line graphs. The variation of the indicated volatile content due to oxidation is independent of the nature of the coal, but is dependent upon the time of heating and the sort of crucible employed, each form of crucible having a constant oxidation constant. The loss of weight during prolonged heating is not always a sign of oxidation, as a very gradual decomposition of the coal substance may be taking place. In experiments to show the influence of ash content on the amount of volatile determined it was found better to mix the coal with a proportion of its own ash than to add inert foreign substance, as in the tests made by Muck, who used powdered quartz. By the addition of ash the amount of volatile found reckoned on the weight of the coal used, increases. In experiments where quartz powder was mixed with the coal irregular results were obtained. The time for the complete degasification of the sample was found to be less as the proportion of ash mixed with the coal was increased. The addition of inert matter to the coal gives various results with different inert materials, and coke samples of different appearance, which fact may be of importance in estimating the coking value of a coal. In two samples of coal and one of pure carbon, a continuous loss of weight during prolonged heating was observed. The graphs of this loss against time were parallel. H. M.

SHALE RETORT.—W. A. Lamb, Assr. to N. S. Ballantine. U.S.P. 1,471,492 of Oct. 23, 1923. (Appl. Dec. 6, 1920.)

The distillation chamber has a sloping heated floor, provided with transverse depressions. Rotary elements fitted to each depression cause the shale to pass down the sloping floor and also agitate the shale. After passing through the retort the spent shale is delivered to the combustion space, where it supplies heat for the distillation. T. A. S.

TREATMENT OF OIL SHALE.—R. C. Dundas and R. T. Howes, Assrs. to S. E. Co. U.S.P. 1,469,628 of Oct. 2, 1923. (Appl. Dec. 23, 1921.)

Shale is distilled in a long vertical chamber, heat being supplied by the combustion of the shale. Combustion is started at the top of the chamber and regulated by the admission of air, the products of the distillation being drawn off at the bottom of the distillation chamber. T. A. S.

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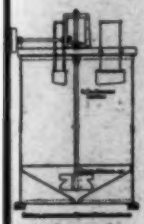

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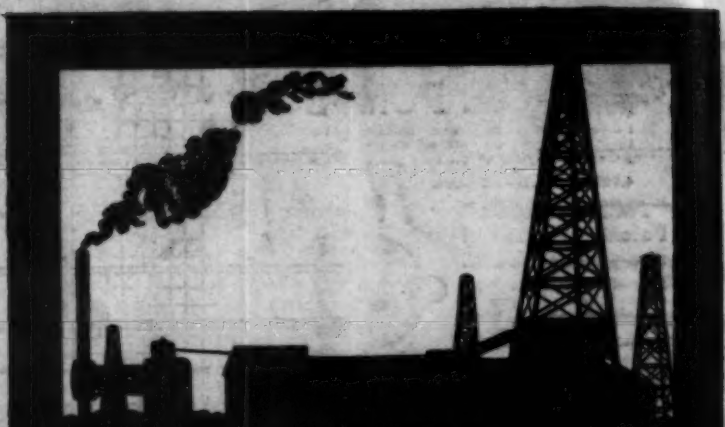

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